











## PRACTICAL AGRICULTURAL CHEMISTRY



# PRACTICAL AGRICULTURAL CHEMISTRY

A MANUAL OF QUALITATIVE AND QUANTITATIVE  
ANALYSIS FOR AGRICULTURAL STUDENTS

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## P R E F A C E

THIS book is intended as a course of laboratory practice for the use of students in agricultural chemistry.

The opening chapters treat on chemical manipulation and apparatus, and the qualitative and quantitative analysis of the most commonly occurring substances with which the student of agriculture must necessarily become practically acquainted with.

The remaining analytical chapters contain various processes for the estimation of the principal constituents of soils, manures, dairy and other agricultural products, and water, my endeavour having been to describe, omitting unnecessary theoretical matter, the most useful, typical, and modern methods in as intelligible a manner and as far as was possible in a volume of this size. The student may therefore apply the processes described to the analysis of many substances, not mentioned here, with which he may hereafter meet, and for this purpose I have devoted a large amount of space to various analytical tables.

F. D. S. R.

GLASGOW,  
*February, 1907.*



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# PRACTICAL AGRICULTURAL CHEMISTRY

## CHAPTER I

### CHEMICAL APPARATUS AND MANIPULATION

**The Balance.**—Fig. 1 illustrates a typical chemical balance, consisting of a beam supported about its centre of gravity by a steel or agate knife-edge, and from the ends of which, suspended by stirrups, working also upon knife-edges, are the scale-pans.

When unoccupied, or when placing on any weight, the beam and pans are raised above their knife-edges by rests, the motion of which is controlled simultaneously by the milled head placed either in front or at the side of the balance.

In weighing, the beam is lowered, the pans being at the same time set free, and the oscillation of the pointer carefully noted upon the ivory scale. The shorter the beam, the more rapid will be the oscillation, and the time occupied in making a weighing thus shortened.

In setting a balance, care must be taken that the table or shelf upon which it stands is fixed as permanently and rigid as possible. The best arrangement is a strong shelf covered with a sheet of thick plate-glass, set dead level, and supported by means of brackets fixed firmly into the wall. If floor connection is avoided, much trouble with vibrations will be overcome.

In choosing a balance, one ought to see that the glass case is well fitted, and that it opens and shuts without any jarring. The balance should be situated in a special room, which may be partitioned off from the laboratory, away from all fumes and dust,

sudden changes of temperature, or the direct rays of the sun. Failing a special room, the balance, with its accessories, weights, etc., may be enclosed in a second and larger glass case, provided with sliding glass sashes.

The balance should be cleaned regularly, only special balance oil being used for lubricating, and the desiccator should be filled whenever necessary with fresh dry calcic chloride. In adjusting a

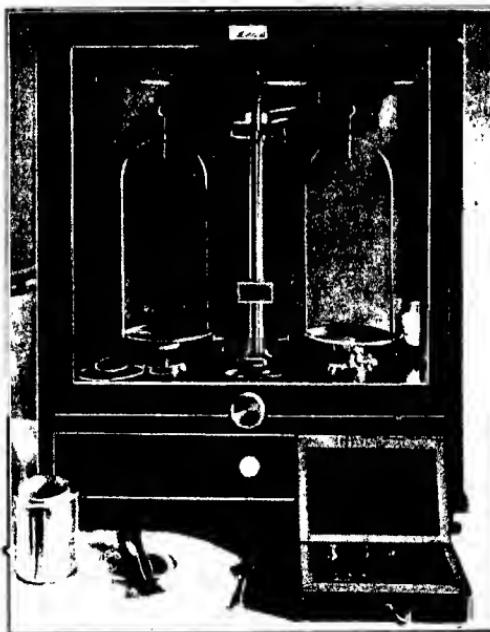


FIG. 1.—CHEMICAL BALANCE: WEIGHTS ON RIGHT

balance after it has been moved, a true level must be obtained by means of the three level screws fixed under the case. In the finer balances the beam is divided either into twelve or ten equal divisions, each of which may again be equally subdivided. With the former graduations a 12-milligramme rider is used, and a 10-milligramme rider must be taken for a beam divided into ten parts.

**Weights.**—The system used in analytical weighing is the metric,

with the exception of, perhaps, in assaying, where the English (grain) weights are sometimes used for convenience in calculating. The weights (1 gramme and upwards) are made of lacquered or plated brass, those smaller than 1 gramme being made either of aluminium or platinum, and are arranged in a suitable box provided with a pair of ivory-tipped forceps for handling them. The weights should be lifted by the forceps only, and retained in their places in the box when not in use.



FIG. 2.—DESICCATORS.

The substances to be weighed should always be placed upon the left pan of the balance, upon a tared watch-glass or other suitable vessel; and in no case should any substance come into contact with the pan itself. It will be found extremely useful to have a small and a large tared watch-glass reserved for this purpose only; a small glass scoop will be found handy in instances where the substance is to be transferred direct into a flask or bottle. For clean dry substances a pair of counterpoised aluminium shallow cups is useful.

The weights should be placed upon the right pan systematically.

It is advisable to commence with a weight which the operator considers is a little too heavy than with one that is too light. Lower weights are then tried, proceeding downwards until the milligrammes and fractions thereof are used by means of the rider weights, these last weights being only used with balance-case closed.

Perfect equilibrium may then be ascertained by the pointer swinging an even number of oscillations to either side of the scale.

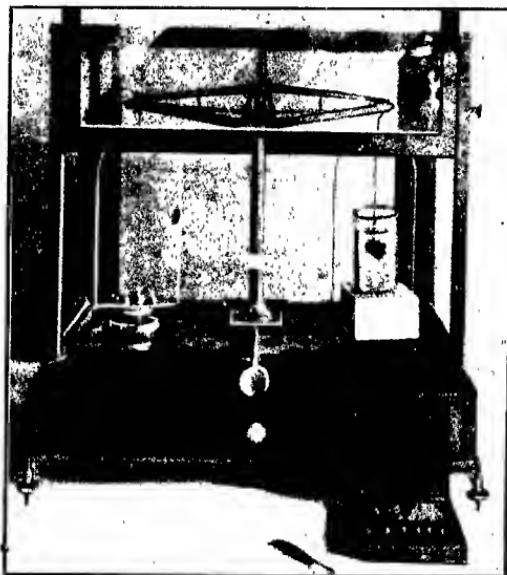


FIG. 3.—SPECIFIC GRAVITY (SUBSTANCE HEAVIER THAN, AND INSOLUBLE IN, WATER).

Before transferring all weights, as well as substances, the balance must be brought to rest and lifted out of action by means of the concentric lever, otherwise the adjustment becomes impaired and the working parts unnecessarily worn.

Immediately a weight is taken the result should be carefully noted, not only as to the weights on the pan, but also by checking such by those weights which are in the box, the amount being noted in the laboratory book.

A warm substance must never be weighed, neither should a very cold substance; it should be allowed to cool in a desiccator to room temperature previous to weighing. Certain substances absorb moisture while being weighed, and that in some cases so rapidly that it is impossible to ascertain their true weight if exposed to the air. Others, again, are volatile or efflorescent. All such bodies must be enclosed in suitable tared weighing vessels, such as a pair of watch-glasses having ground edges and clamped by means of a spring clip, or a thin glass-stoppered weighing-bottle or tube.

**Specific Gravity.** — This is determined at a temperature of  $15\frac{1}{2}^{\circ}$  C., this being the normal temperature of a room, and accordingly the measuring vessels and weights are calibrated and adjusted at this temperature.

In ascertaining the specific gravity of solids heavier than water, and insoluble in that liquid, the substance is suspended by a fine thread or fibre from the left-hand end of the beam of a balance, and its weight when in air is taken. A small wooden trestle, such as are supplied with balances, is then placed over the pan, and supports a small beaker of distilled water. The substance is then just immersed in the water, all adhering bubbles of air being removed by means of a brush, and the weight is again taken.

Then if  $W$  = Weight of solid in air,  
 $W^*$  = Weight of solid in water,

The specific gravity of the substance will be  $\frac{W}{W - W^*}$ .

When the substance is lighter than, but insoluble in, water, its weight in air is ascertained as already described, and it is then immersed in water with the aid of a "sinker" attached to it. The weight of the "sinker" alone in water is also taken.

Then if  $W$  = Weight of substance in air,  
 $X$  = Loss of weight in water,  
 $S$  = Weight of "sinker" in water,  
 $C$  = Weight of substance plus "sinker"  
 in water.

The specific gravity of the substance will be :

$$\frac{W}{X} = \frac{W}{W - (C - S)} = \frac{W}{W - (C - S)}$$

When the substance is soluble in water, it is weighed in air, and

then in a liquid in which it is insoluble, the specific gravity of the liquid used being also ascertained.

Then if  $W$  = Weight of substance in air,  
 $W'$  = Weight of substance in the liquid,  
 $X$  = Specific gravity of liquid,

The specific gravity of the substance compared to the liquid is

$$\frac{W}{W - W'}$$

The specific gravity of the liquid : specific gravity of water ::  $X : 1$ .

Therefore the specific gravity of the substance related to that of water will be  $\frac{W}{W - W'} \times X$ .

If the substance is in the state of a fine powder it may be introduced into a specific-gravity bottle, and its weight in the bottle ascertained by weighing in air and then in water, the weight of water required to fill the bottle being also obtained.

Then if  $W$  = Weight of solid,  
 $W_1$  = Weight of solid plus water,  
 $W_2$  = Weight of water required to fill the bottle,

The specific gravity of the substance—

$$\frac{W}{W - (W_1 - W_2)} = \frac{W}{W + W_2 - W_1}$$

The specific gravity of a liquid may be ascertained by four methods—(1) By the Sprengel tube; (2) by the Westphal balance; (3) by the hydrometer; (4) and most accurately by the specific-gravity bottle.

The Sprengel tube consists in a thin-walled glass U-tube having its open ends drawn out to a capillary bore and bent at right angles. The capillaries are marked by means of a light file near their open ends. The tube is filled with distilled water by introducing one end into a beaker of the water and sucking in the liquid by applying the mouth or a sucker to the other end. The tube, when held upright in the balance by a thread, should contain just as much of the liquid as shall reach both marks; any excess must be carefully wiped off with a piece of filter paper. The weight is then taken. The weight of the liquid under examination is next observed in a

similar manner, and after drying the tube, by dividing the weight of the substance by the weight of water the specific gravity is obtained.

The Westphal balance is here illustrated, and is simply a variety of balance in which the weights are hung upon a graduated beam, the plummet being immersed in the liquid under examination, after having first and for all been adjusted by immersion in distilled water at  $15^{\circ}$  C. The weights employed indicate the specific gravity of the fluid direct.

The hydrometer is a glass or metal float, weighted with mercury in such a manner as to float upright in the liquid. The stem is graduated, and the reading is taken upon the stem at the surface of

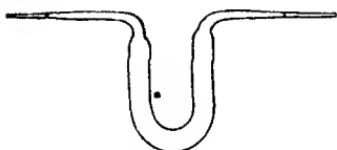


FIG. 4.—SPRENGEL TUBE.

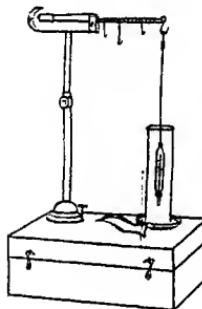


FIG. 5.—WESTPHAL BALANCE.

the still liquid when the instrument is stationary. Hydrometers are graduated in various degrees or calibrations to suit special purposes.

The specific-gravity bottle is a small, thin, glass-stoppered bottle, usually of 25, 50, or 100 cubic centimetres capacity. The neck is somewhat contracted in its descent, and in this way the stopper, which is perforated, is held more securely. The flask is filled to the top, the stopper inserted quickly, and in such a way that the capillary bore is filled with the fluid; the excess drops on the top of the stopper, being immediately removed by wiping with the palm of the hand. A cloth must never be used for this purpose, as its contact would draw up too much of the liquid from the

capillary bore by capillary attraction. The exterior of the bottle itself, however, may be dried with a clean cloth. If the temperature of the liquid is below  $15.5^{\circ}\text{C}.$ , it must be raised to that degree by gently warming, the exact temperature being observed by inserting a thermometer into the bottle and stirring constantly. If the liquid

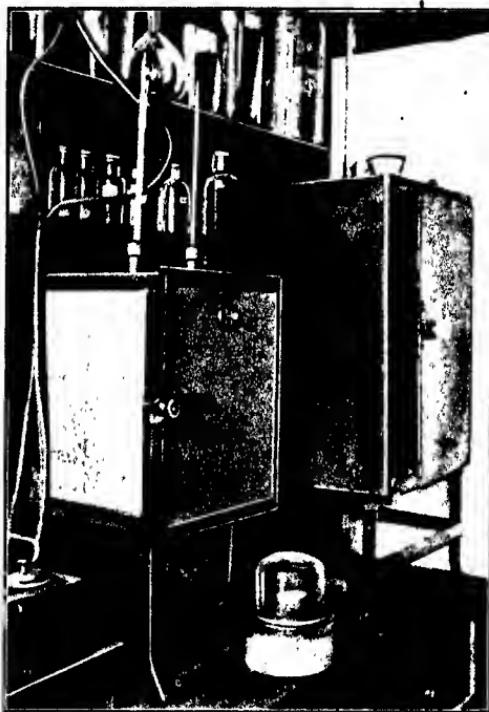


FIG. 6.—AIR AND WATER OVENS.

is too warm it may be cooled in iced water, or water containing crystals of ammonium chloride.

The weight of the clean dry bottle and its weight when filled with distilled water having been ascertained, the exact weights of the amounts of water and liquid respectively required to fill the bottle are obtained by subtraction.

Then, specific gravity of liquid =

Weight of liquid required to fill bottle.

Weight of water required to fill bottle.

- **Besiccation.**—Substances in which the moisture is to be estimated, or which require to be dried—*e.g.*, precipitates—are usually dried in the copper, water or hot-air oven. Water-ovens are made in all sizes and shapes to fit any given requirements, and may be had with constant level water-supply. As substances when in the dry or hot condition readily absorb moisture from the atmosphere during cooling, they must be placed in a desiccator whilst they cool. This vessel (Fig. 2) is one in which a dry atmosphere is

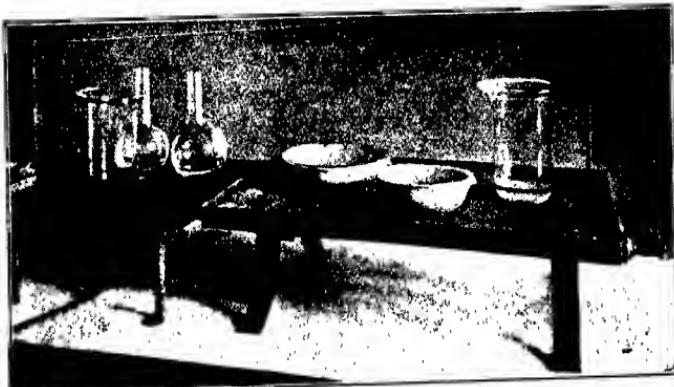


FIG. 7.—SAND-BATH AND HOT-PLATE.

- maintained by means of the presence of some desiccating agent—*e.g.*, strong sulphuric acid or calcium chloride. The latter substance is preferable, as splashing is avoided. If acid must be used, it will be found advantageous to mix it with a little dry sand sufficient to form a paste. Solutions which require to be evaporated may be placed in porcelain, glass, platinum, or nickel vessels, and heated over a water or sand bath or hot iron plate heated by a radial burner.

For very slow evaporation, especially at a temperature just below the boiling-point, or to avoid spitting, a water-bath is advisable.

Solution may be accelerated by heating over a Bunsen burner, either with or without wire gauze or a sheet of asbestos. To avoid loss by spilling, the dish or beaker may be covered by a glass. If a flask, it may be slightly inclined or a small filter-funnel inserted in its neck. Violent ebullition may be avoided by adding a small piece of platinum foil or coiled wire or a piece of clean ignited pipe-clay or pumice.



FIG. 8.—WATER-BATH WITH EVAPORATING BASIN PROTECTED WITH CLOCK GLASS.

The Victor Meyer funnel (Fig. 9) will be found very convenient when a long evaporation is in process in hastening the operation by retaining the steam in such a way as to form a jacket, and in protecting the dish from the dust of the laboratory.

**Precipitation and Filtration.**—In quantitative analysis precipitation must be complete, and in order to effect this the solution must either be boiled or well stirred, as the case permits, with the precipitant. In hot precipitation complete reaction is observed

by a rapid separation of the precipitate. Some substances—*e.g.*, magnesium ammonium phosphate—require time, together with thorough agitation in a cold solution. Care should be taken to avoid the use of excessive quantities of reagents in all cases, and the filtrate must always be tested by the addition of a further small quantity of the precipitating reagent. All filter-funnels should be



FIG. 9.—WATER-BATH AND VICTOR MEYER FUNNEL.

carefully picked out and tested as to their rapidity by pressing the filter-paper lightly and evenly to the sides, and passing through a measured quantity of water. The quick filters should then be marked on their rim by a file. The filter-paper should not completely fill the funnel, and it must never be filled to within less than  $\frac{1}{4}$  inch from its edge with the solution. If a plain beaker is

used in preference to a spouted one, its outer edge may be smeared with a little vaseline or hot indiarubber ; this prevents spilling. The solution must be guided on to the filter with the aid of a glass rod tipped with rubber (a "policeman"), unless in cases where the rubber is liable to be attacked by the solution.

It is advisable to allow the precipitate to settle first. The clear liquid is then run through the filter by careful decantation, and the precipitate washed by decantation and transferred by the aid of the glass rod, together with a small quantity of the washing solution. Any adhering particles are also rubbed off and transferred to the filter, and finally the vessel is carefully rinsed out several times with the washing solution. The precipitate in the filter is then washed as may be directed, always taking care to allow each washing to drain completely away before adding more. Evaporate a small portion of the final washings on a thin glass to see that all dissolved salts are washed through.

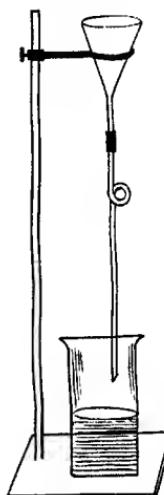


FIG. 10.—ACCELERATED FILTRATION (LENGTHENED FUNNEL-TUBE).

**Filter-Pumps.**—Slow filtration may be accelerated by extending the stem of the funnel by means of a long tube. In obstinate cases, however, as in gelatinous precipitates, recourse must be had to the filter-pump. Before placing the filter in the funnel a small perforated platinum cone or disc is introduced. This gives support to the paper, and prevents bursting. The funnel is then passed through a rubber core, and firmly fixed in a filter-flask which is attached by non-collapsible tubing to the filter-pump. The exhaust must be turned on slowly, and the filter should not be allowed to run quite dry. The precipitate is then covered with a paper and dried in the oven. Small precipitates may be detached from the funnel and dried in their filters upon dishes. This hastens the operation.

The dried precipitate may then be weighed in a tared weighing-bottle, in which case the weight of the paper must have been previously ascertained, or it may be ignited in a porcelain or

platinum vessel over a Bunsen, Argand, or blowpipe burner, or in a muffle furnace, as may be directed. The latter arrangement is very convenient in cases where a high temperature for many samples is required.

**Ignition of Precipitates.**—For general agricultural analyses a thin platinum capsule about 2 inches wide by 1 inch deep will be found most convenient for igniting most precipitates in. Platinum utensils, when repeatedly heated, tend to become blistered, and show small hair cracks. These are best removed from time to



FIG. 11.—ACCELERATED FILTRATION (FILTER-FLASK, ETC.).

time by gently rubbing with a little moist silver sand. In this way a nice polish is sustained, and the life of the platinum prolonged. Obstinate stains may be removed by gently fusing with a little acid potassium sulphate, and finally rubbing up with a little silver sand. Porcelain crucibles, when badly burnt, may be cleaned by fusing with a little fusion mixture and subsequent treatment with a little hydrochloric acid. This tends, however, to wear away the enamel. Ignition vessels should be cleaned and weighed previously to each operation. The incineration of the filter and contents may

be proceeded with immediately it is dry. The precipitate is carefully detached from the filter, spread out upon a piece of glazed paper or tile, by the aid of a thin, stiff spatula and a fine camel's-hair brush. The filter is then folded and held by the rim by a pair of tongs or forceps, the apex being downwards, and ignited. As soon as the last glow has vanished it is dropped into the crucible, the lid is put on, and it is ignited over a burner until the ash is white. Filters which are slow to ignite may be drenched with a few drops of ammonium nitrate, carefully dried, and ignited as before. The filter having been properly ashed, the precipitate is then added, and both are again heated as required. All crucibles should be allowed to cool thoroughly in the desiccator before being weighed. This generally occupies about twenty minutes.

In igniting a wet filter and contents together, it should be allowed to drain as much as possible, and placed in the capsule, and heat applied very gently at first to avoid spitting.

**Gooch Treatment of Precipitates.**—A rapid and convenient treatment method of dealing with precipitates is that involving the use of the Gooch crucible. In this device the solution is filtered through a layer either of prepared paper or clean asbestos. The latter may be prepared by scraping to fine short down, white silky asbestos, boiling it with hydrochloric acid to remove soluble matter, and then well washing it with water by decantation. A bottle full of this dried material should be prepared at a time, from which requisite quantities may be taken as required. The Gooch crucible, which is usually of platinum or porcelain, has a perforated bottom, and is fitted tightly into a filter-funnel placed in a filter-flask. A little of the prepared asbestos is spread over the bottom of the crucible in an even layer, and a little water added. On the vacuum being started, the water passes through, leaving the asbestos in a compact layer upon the perforated bottom. The filtration of a precipitate may now be proceeded with in the usual manner, when it will be found that the operation is much more accelerated. The precipitate is dried and ignited *in situ* in the crucible.

**Bumping of Boiling Liquids.**—In most cases this is remedied by the addition of a piece of platinum foil or coil of wire or a piece of pumice or pipe-clay. In more difficult cases—e.g., in distilling methylic alcohol—the introduction of a small piece of sodium tin

or amalgam will be found satisfactory. When the presence of extraneous substances is impermissible, a glass tube drawn out to a capillary tube may be passed through a cork in such a manner that its capillary end presses gently against the bottom of the

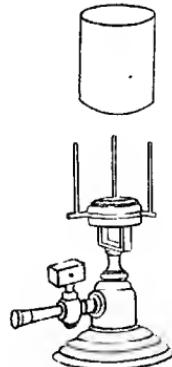


FIG. 12.—ARGAND  
BURNER.

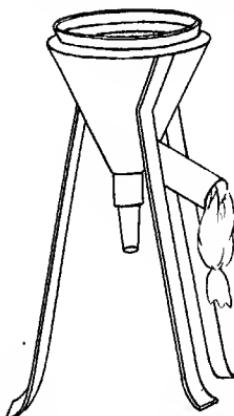


FIG. 12.—HOT-WATER FILTER-  
FUNNEL.

vessel. By this means a slow current of air, carbon dioxide, or other gas, may be passed through the liquid when boiling.

**Accelerated Hot Filtration.**—The student will find that hot liquids filter more rapidly than cold ones; when it is necessary to maintain a convenient temperature—e.g., in filtering fats—the liquid may be passed through a hot-water jacketed funnel.

## CHAPTER II

### QUALITATIVE ANALYSIS

THE more common metals with which the student must first become acquainted are arranged in the following five groups :

- Group 1.*—Those in which the chlorides are insoluble in water.
- Group 2.*—Those in which the sulphides are insoluble in water.
- Group 3.*—Those in which (*a*) the hydrates, (*b*) the sulphides, are insoluble in water.
- Group 4.*—Those in which the carbonates are insoluble in water.
- Group 5.*—Those which require separate individual tests.

#### Classification of the Commoner Metals.

Group	Metal.	Reagent.	Nature of Precipitate.
1	Pb"	HCl	PbCl <sub>2</sub> ; white.
	Ag	HCl	AgCl; white.
	Hg"	HCl	HgCl <sub>2</sub> ; white.
2	Pb		PbS; black; insoluble in (NH <sub>4</sub> ) <sub>2</sub> S
	Hg		HgS; black; " "
	Bi	H <sub>2</sub> S	Bi <sub>2</sub> S <sub>3</sub> ; black; " "
	Cu		CuS; black; " "
	Cd	(In solution slightly acid with HCl)	CdS; yellow; " "
	As		As <sub>2</sub> S <sub>3</sub> ; yellow; soluble in (NH <sub>4</sub> ) <sub>2</sub> S*
	Sb		Sb <sub>2</sub> S <sub>3</sub> ; orange; " "
	Sn		SnS; brown; " "
			SnS <sub>2</sub> ; yellow; " "
3	Fe		Fe(OH) <sub>2</sub> ; dirty green.
	Al	NH <sub>4</sub> Cl + NH <sub>4</sub> OH	Fe <sub>2</sub> (OH) <sub>3</sub> ; red.
	Cr		Al <sub>2</sub> (OH) <sub>5</sub> ; white.
	Mn		Cr <sub>2</sub> (OH) <sub>5</sub> ; green.
	Zn		MnS; flesh colour.
	Co	(NH <sub>4</sub> ) <sub>2</sub> S	ZnS; white.
	Ni		CoS; black.
			NiS; black.

## Classification of the Commoner Metals—continued.

Group.	Metal.	Reagent.	Nature of Precipitate.
4	Ba	$\text{NH}_4\text{Cl} + \text{NH}_4\text{OH} + (\text{NH}_4)_2\text{CO}_3$	$\text{BaCO}_3$ ; white.
	Sr		$\text{SrCO}_3$ ; white.
	Ca		$\text{CaCO}_3$ ; white.
5	Mg	$\text{NH}_4\text{OII} + \text{Na}_2\text{HPO}_4$	$\text{MgNH}_4\text{PO}_4$ ; white.
	K	$\text{PtCl}_4$	$\text{K}_2\text{PtCl}_6$ ; yellow crystalline.
	Na		$\text{Na}_2\text{PtCl}_6$ ; yellow crystalline.
	$\text{NH}_4$	$\text{HgI}_2 \cdot 2\text{KI}$	$\text{Hg}_2\text{NI}$ ; yellow to brown.

## Reactions of the Common Metals.

Lead (*ous*),  $\text{Pb}^+$ .

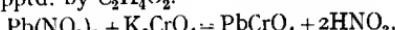
1. HCl gives a white ppt. of  $\text{PbCl}_2$ ; sol. in hot water, insol. in  $\text{NH}_4\text{OH}$ .



2.  $\text{H}_2\text{S}$  gives a black ppt. of  $\text{PbS}$ ; sol. in dilute hot  $\text{HNO}_3$ .



3.  $\text{K}_2\text{CrO}_4$  gives a yellow ppt. of  $\text{PbCrO}_4$ ; sol. in  $\text{NaOH}$ , and  
reptd. by  $\text{C}_2\text{H}_4\text{O}_2$ .



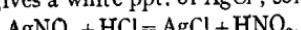
4.  $\text{H}_2\text{SO}_4$  gives a white ppt. of  $\text{PbSO}_4$ ; sol. in  $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$  or  $\text{NaOH}$ .

Lead (*ic*),  $\text{Pb}^+$ .

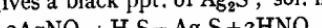
1.  $\text{H}_2\text{S}$  gives a black ppt. of  $\text{PbS}$  even in a HCl solution.

## Silver, Ag.

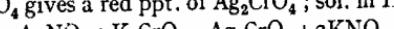
1. HCl gives a white ppt. of  $\text{AgCl}$ ; sol. in  $\text{NH}_4\text{OH}$ .



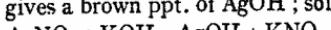
2.  $\text{H}_2\text{S}$  gives a black ppt. of  $\text{Ag}_2\text{S}$ ; sol. in dilute  $\text{HNO}_3$ .



3.  $\text{K}_2\text{CrO}_4$  gives a red ppt. of  $\text{Ag}_2\text{CrO}_4$ ; sol. in  $\text{HNO}_3$ .

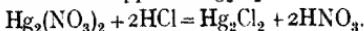


4. KOH gives a brown ppt. of  $\text{AgOH}$ ; sol. in  $\text{NH}_4\text{OH}$ .

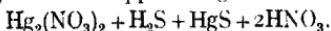


**Mercury (ous), Hg'.**

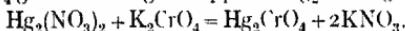
1. HCl gives a white ppt. of  $\text{Hg}_2\text{Cl}_2$ .



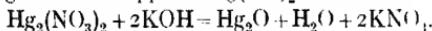
2.  $\text{H}_2\text{S}$  gives a black ppt. of  $\text{HgS}$ .



3.  $\text{KCrO}_4$  gives an orange-red ppt. of  $\text{Hg}_2\text{CrO}_4\text{Hg}^{(+)}$ .



4. KOH gives a black ppt. of  $\text{Hg(OII)}_2$ .

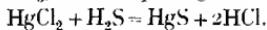


5.  $\text{SnCl}_2$  gives a grey-white ppt. of metallic Hg.

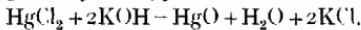
**Mercury (ic), Hg".**

1. HCl gives no ppt.

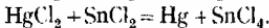
2.  $\text{H}_2\text{S}$  gives a black ppt. of  $\text{HgS}$  : insol. in  $(\text{NH}_4)_2\text{S}$  or hot  $\text{HNO}_3$ , sol. in aqua regia.



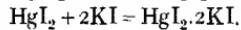
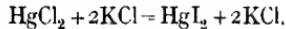
3. KOH gives a yellow ppt. of  $\text{HgO}$  : insol. in excess.



4.  $\text{SnCl}_2$  gives a white ppt. of  $\text{Hg}_2\text{Cl}_2$ , which is changed to grey metallic Hg in excess.



5. KI gives a vermillion ppt. of  $\text{HgI}_2$  ; sol. in excess to a clear sol. and in KOH.

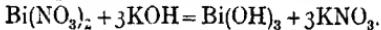
**Bismuth, Bi.**

1. HCl gives no ppt.

2.  $\text{H}_2\text{S}$  gives a black ppt. of  $\text{Bi}_2\text{S}_3$  ; insol. in  $(\text{NH}_4)_2\text{S}$ , sol. in hot dil.  $\text{HNO}_3$ .



3. An alkaline hydrate—e.g., KOH—gives a white ppt. of  $\text{Bi}(\text{OH})_3$  ; sol. in acids.



**Copper, Cu.**

1. HCl gives no ppt.
2.  $\text{H}_2\text{S}$  gives a black ppt. of  $\text{CuS}$ ; insol. in  $(\text{NH}_4)_2\text{S}$ , or hot dil.  $\text{H}_2\text{SO}_4$ , sol. in hot  $\text{HNO}_3$  to a blue sol.  

$$\text{CuSO}_4 + \text{H}_2\text{S} = \text{CuS} + \text{H}_2\text{SO}_4.$$
3.  $\text{NH}_4\text{OH}$  gives a greenish-blue ppt. of  $\text{CuSO}_4 \cdot 2\text{Cu}(\text{OH})_2$  sol. in excess to a dark blue sol.  

$$3\text{CuSO}_4 + 4\text{NH}_4\text{OH} = \text{CuSO}_4 \cdot 2\text{Cu}(\text{OH})_2 + 2(\text{NH}_4)_2\text{SO}_4.$$
4. KOH or NaOH gives a pale blue ppt. of  $\text{Cu}(\text{OH})_2$ ; insol. in excess and blackening on heating.  

$$\text{CuSO}_4 + 2\text{KOH} = \text{Cu}(\text{OH})_2 + \text{K}_2\text{SO}_4.$$
5.  $\text{K}_4\text{Fe}(\text{CN})_6$  gives a reddish-brown ppt. of  $\text{Cu}_2\text{Fe}(\text{CN})_6$ .  

$$2\text{CuSO}_4 + \text{K}_4\text{Fe}(\text{CN})_6 = \text{Cu}_2\text{Fe}(\text{CN})_6 + 2\text{K}_2\text{SO}_4.$$
6. Most copper salts, especially the chlorides, burn with a bright green coloured flame.

**Cadmium, Cd.**

1. HCl gives no ppt.
2.  $\text{H}_2\text{S}$  gives a yellow ppt. of  $\text{CdS}$ ; insol. in  $(\text{NH}_4)_2\text{S}$ , sol. in hot dil. HCl or  $\text{HNO}_3$ .  

$$\text{Cd}(\text{NQ}_3)_2 + \text{H}_2\text{S} = \text{CdS} + 2\text{HNO}_3.$$
3. KOH or NaOH gives a white ppt. of  $\text{Cd}(\text{OH})_2$ ; insol. in excess.  

$$\text{Cd}(\text{NO}_3)_2 + 2\text{KOH} = \text{Cd}(\text{OH})_2 + 2\text{KNO}_3.$$
4.  $\text{NH}_4\text{OH}$  gives a white ppt. of  $\text{Cd}(\text{OH})_2$ ; sol. in excess.  

$$\text{Cd}(\text{NO}_3)_2 + 2\text{NH}_4\text{OH} = \text{Cd}(\text{OH})_2 + 2\text{NH}_4\text{OH}.$$

**Arsenic, As.**

1. HCl gives no ppt.
2.  $\text{H}_2\text{S}$  in weak HCl sol. gives a yellow ppt. of  $\text{As}_2\text{S}_3$ ; sol. in •  $(\text{NH}_4)_2\text{S}$ .  

$$2\text{Na}_3\text{AsO}_4 + 6\text{HCl} + 2\text{H}_2\text{S} = 2\text{H}_3\text{AsO}_4 + 6\text{NaCl} + 2\text{H}_2\text{O} + \text{S}_2$$
  

$$2\text{H}_3\text{AsO}_4 + 2\text{H}_2\text{S} = \text{As}_2\text{S}_3 + 6\text{H}_2\text{O}.$$
3.  $\text{AgNO}_3$  gives a yellow or red ppt. of  $\text{Ag}_3\text{AsO}_4$ ; sol. in  $\text{NH}_4\text{OH}$  and in acids.  

$$\text{Na}_3\text{AsO}_4 + 3\text{AgNO}_3 = \text{Ag}_3\text{AsO}_4 + 3\text{NaNO}_3.$$
  

$$\text{Na}_3\text{AsO}_4 + 3\text{AgNO} = \text{Ag}_3\text{AsO}_4 + 3\text{NaNO}_3.$$

4. *Marsh's Test.*—Nascent hydrogen is generated in an acid solution of arsenic by the introduction of a piece of arsenic-free zinc, whereby  $\text{AsH}_3$  is evolved. The gas may be passed through a red-hot tube and decomposed into metallic arsenic, which deposits in the colder portion (sublimation) as a brown or dark grey mirror, and hydrogen; or the gas may be ignited, when it burns with a lavender-coloured flame, forming  $\text{As}_2\text{O}_3$ . If a piece of cold porcelain be now held in the flame, incomplete combustion is formed, and the arsenic is deposited as a dark brown stain on the slab. This stain is soluble in a freshly prepared aqueous solution of bleaching-powder ( $\text{CaClOCl}$ ) (contrast antimony). If the gas is passed through a solution of  $\text{AgNO}_3$  or through a filter-paper saturated with the same (Gutzeit test), a yellow precipitate or stain of  $\text{Ag}_3\text{AsO}_3$  will be formed.
5. *Reinsch's Test.*—The solution of arsenic is effected in HCl to which a bright strip of pure electrolytic copper foil is added, and the whole is gently boiled for a few minutes. The arsenic is deposited as a steel-grey film upon the copper. The foil is then dried, weighed, and heated in a dry test-tube or Berzelius tube. The arsenic is sublimed upon the colder portion of the tube in the form of dull steel-grey octahedric crystals (contrast antimony) of  $\text{As}_2\text{O}_3$ .

### **Antimony, Sb.**

1. To a HCl solution of antimony a piece of zinc and a piece of platinum foil are added, both being kept in touch with each other. The antimony is deposited after some little time upon the platinum as a black stain.
2. *Marsh's Test.*—This test is carried out as for arsenic (*q.v.*). The antimony forms an amorphous deposit, insoluble in a freshly prepared solution of bleaching-powder. Antimoniated hydrogen ( $\text{Sb}_2\text{S}_3$ ) produces a black stain or precipitate with silver nitrate of  $\text{SbAg}_3$ .
3. HCl gives no ppt.

4.  $\text{H}_2\text{S}$  gives an orange ppt. of  $\text{Sb}_2\text{S}_3$ ; sol. in  $(\text{NH}_4)_2\text{S}$  or hot  $\text{HCl}$ , insol. in  $(\text{NH}_4)_2\text{CO}_3$ .  

$$2\text{SbCl}_3 + 3\text{H}_2\text{S} = \text{Sb}_2\text{S}_3 + 6\text{HCl}.$$

$$\text{Sb}_2\text{S}_3 + 3(\text{NH}_4)_2\text{S} = 2(\text{NH}_4)_2\text{SbS}_3.$$
5.  $\text{KOH}$  gives a white ppt. of  $\text{Sb}_2\text{O}_3$ ; sol. in excess.  

$$2\text{SbCl}_3 + 3\text{KOH} = \text{Sb}_2\text{O}_3 + 3\text{KCl} + 3\text{HCl}.$$

$$\text{Sb}_2\text{O}_3 + 2\text{KOH} = \text{K}_2\text{Sb}_2\text{O}_4 + \text{H}_2\text{O}.$$
6.  $\text{NH}_4\text{OH}$  gives a white ppt. of  $\text{Sb}_2\text{O}_3$ ; insol. in excess.
7.  $\text{H}_2\text{O}$  gives a white ppt. of  $\text{SbOCl}$ ; sol. in tartaric acid (contrast Bi).  

$$3\text{SbCl}_3 + 3\text{H}_2\text{O} = 3\text{SbOCl} + 6\text{HCl}.$$
8.  $\text{AgNO}_3$  gives in an alkaline sol. a black ppt.

**Tin, Sn.**

1.  $\text{HCl}$  gives no ppt.
2.  $\text{H}_2\text{S}$  gives a yellow to brown ppt. of  $\text{SnS}_2$  to  $\text{SnS}$ ; sol. in  $(\text{NH}_4)_2\text{S}$  or  $\text{NaOH}$ , from which it is repptd. by  $\text{HCl}$ ; sol. in  $\text{HCl}$ , insol. in  $(\text{NH}_4)_2\text{CO}_3$ .  

$$\text{SnCl}_2 + \text{H}_2\text{S} = \text{SnS} + 2\text{HCl}.$$

$$\text{SnS} + (\text{NH}_4)_2\text{S} = (\text{NH}_4)_2\text{S} \cdot \text{SnS}.$$

$$\text{SnCl}_2 + 2\text{H}_2\text{S} = \text{SnS} + 4\text{HCl}.$$
3.  $\text{HgCl}_2$  gives a white ppt. of  $\text{Hg}_2\text{Cl}_2$ , becoming grey on heating ( $\text{Hg}$ ).  

$$\text{SnCl}_2 + \text{HgCl}_2 = \text{Hg}_2\text{Cl}_2 + \text{SnCl}_4.$$

$$\text{Hg}_2\text{Cl}_2 + \text{SnCl}_2 = \text{Hg}_2 + \text{SnCl}_4.$$

$$\text{SnCl}_4 + \text{HgCl}_2 = \text{no change.}$$

**Iron (ous), Fe.**

1.  $\text{HCl}$  gives no ppt.
2.  $\text{H}_2\text{S}$  gives no ppt. in acid sol.
3.  $\text{NH}_4\text{OH}$  gives a white ppt. of  $\text{Fe(OH)}_2$ , rapidly turning from green to brown.  

$$\text{FeSO}_4 + 2\text{NH}_4\text{OH} = \text{Fe(OH)}_2 + (\text{NH}_4)_2\text{SO}_4.$$
4.  $\text{K}_3\text{FeCy}_6$  gives a dark blue (Turbull's blue) ppt. of  $\text{Fe}_3(\text{FeCy}_6)_2$ .  

$$3\text{FeSO}_4 + 2\text{K}_3\text{FeCy}_6 = \text{Fe}_3(\text{FeCy}_6)_2 + 3\text{K}_2\text{SO}_4.$$

5.  $K_4FeCy_6$  gives a bluish-white ppt. of  $FeK_2FeCy_6$ .  
 $FeSO_4 + K_4FeCy_6 = FeK_2FeCy_6 + K_2SO_4$ .
6. KCNS gives no coloration (contrast ferric salt).
7.  $(NH_4)_2S$  gives a black ppt. of  $FeS$ ; sol. in HCl.  
 $FeSO_4 + (NH_4)_2S = FeS + (NH_4)_2SO_4$ .

### Iron (*ic*), Fe.

1. HCl gives no ppt.
2.  $H_2S$  gives no ppt. in acid sol.
3.  $NH_4OH$  gives a reddish-brown ppt. of  $Fe_2(OH)_6$ ; insol. in excess or in  $NH_4Cl$ , sol. in HCl.  
 $Fe_2Cl_6 + 6NH_4OH = Fe_2(OH)_6 + 6NH_4Cl$ .
4.  $K_4FeCy_6$  gives a blue ppt. (Prussian blue) of  $Fe_4(FeCy)_6$ ; insol. in HCl, sol. in  $H_4C_2O_4$ .  
 $Fe_2Cl_6 + 3K_4FeCy_6 = Fe_4(FeCy)_6 + 12KCl$ .
5.  $K_3FeCy_6$  gives a greenish-brown solution.
6. KCNS gives a blood-red solution of  $Fe_2(CyS)_6$ .  
 $Fe_2Cl_6 + 6KCNS = Fe_2(CNS)_6 + 6HCl$ .
7.  $(NH_4)_2S$  gives a black ppt. of  $FeS$ ; sol. in HCl.

### Chromium, Cr.

1. HCl gives no ppt.
2.  $H_2S$  gives no ppt. in acid sol.
3.  $NH_4OH$  gives a greenish-blue ppt. of  $Cr_2(OH)_3$ ; insol. in excess, sol. in hot HCl.  
 $Cr_2Cl_6 + 6NH_4OH = Cr_2(OH)_3 + 6NH_4Cl$ .
4. NaOH gives a similar ppt.; sol. in excess, and forming  $Cr_2O_3$  on boiling.  
 $Cr_2Cl_6 + 6NaOH = Cr_2(OH)_6 + 6NaCl$ .  
 $Cr_2(OH)_6 + \text{heat} = Cr_2O_3 + 3H_2O$ .
5.  $(NH_4)_2S$  gives a greenish-blue ppt. of  $Cr_2(OH)_3$ , with evolution of  $H_2S$ ; sol. in HCl.

### Aluminium, Al.

1. HCl gives no ppt.
2.  $H_2S$  gives no ppt. in acid sol.

3.  $\text{NH}_4\text{OH}$  gives a white gelatinous ppt. of  $\text{Al}_2(\text{OH})_3$ ; insol. in excess or  $\text{NH}_4\text{Cl}$ .  

$$\text{Al}_2(\text{SO}_4)_3 + 6\text{NH}_4\text{OH} \rightleftharpoons \text{Al}_2(\text{OH})_3 + 3(\text{NH}_4)_2\text{SO}_4.$$
- 4. KOH or NaOH gives a similar ppt.; sol. in excess, not repptd. on boiling, repptd. by  $\text{NH}_4\text{Cl}$ .  

$$\text{Al}_2(\text{SO}_4)_3 + 6\text{KOH} \rightleftharpoons \text{Al}_2(\text{OH})_3 + 3\text{K}_2\text{SO}_4.$$
  

$$\text{Al}_2(\text{OH})_3 + 6\text{KOH} \rightleftharpoons 3\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 + 6\text{H}_2\text{O}.$$
  

$$3\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 + 6\text{NH}_4\text{Cl} \rightarrow \text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O} + 6\text{KCl} + 6\text{NH}_3.$$
5.  $\text{Na}_2\text{HPO}_4$  gives a white ppt. of  $\text{Al}_2\text{PO}_4$ ; sol. in NaOH, sol. in HCl, repptd. by  $\text{NH}_4\text{OH}$  in excess, insol. in  $\text{NH}_4\text{Cl}$  or  $\text{C}_2\text{H}_4\text{O}_2$ .
6.  $(\text{NH}_4)_2\text{S}$  gives a fairly white ppt. of  $\text{Al}_2(\text{OH})_3$ ; sol. in HCl.

**Zinc, Zn.**

1. HCl gives no ppt.
2.  $\text{H}_2\text{S}$  gives no ppt. in acid sol. except in  $\text{C}_2\text{H}_4\text{O}_2$ ; in alkaline sols. it gives a white ppt. of  $\text{ZnS}$ .  

$$\text{ZnSO}_4 + (\text{NH}_4)_2\text{S} \rightleftharpoons \text{ZnS} + (\text{NH}_4)_2\text{SO}_4.$$
  

$$\text{ZnS} + 2\text{HCl} \rightleftharpoons \text{ZnCl}_2 + \text{H}_2\text{S}.$$
3.  $(\text{NH}_4)_2\text{S}$  gives a white ppt. of  $\text{ZnS}$ ; sol. in HCl, insol. in  $\text{C}_2\text{H}_4\text{O}_2$ .  

$$\text{ZnSO}_4 + (\text{NH}_4)_2\text{S} \rightleftharpoons \text{ZnS} + (\text{NH}_4)_2\text{SO}_4.$$
  

$$\text{ZnS} + 2\text{HCl} \rightleftharpoons \text{ZnCl}_2 + \text{H}_2\text{S}.$$
4. KOH or NaOH gives a white ppt. of  $\text{Zn}(\text{OH})_2$ ; sol. in excess.  

$$\text{ZnSO}_4 + 2\text{KOH} \rightleftharpoons \text{Zn}(\text{OH})_2 + \text{K}_2\text{SO}_4.$$
  

$$\text{Zn}(\text{OH})_2 + 2\text{KOH} \rightleftharpoons \text{KO}_2 \cdot \text{ZnO} + 2\text{H}_2\text{O}.$$

**Manganese, Mn.**

1. HCl gives no ppt.
2.  $\text{H}_2\text{S}$  gives no ppt. in acid sols.; in alkaline sols. it gives a flesh-coloured ppt. of  $\text{MnS}$ .  

$$\text{MnCl}_2 + (\text{NH}_4)_2\text{S} \rightleftharpoons \text{MnS} + 2\text{NH}_4\text{Cl}.$$
3.  $(\text{NH}_4)_2\text{S}$  gives a buff or flesh coloured ppt. of  $\text{MnS}$ ; sol. in  $\text{C}_2\text{H}_4\text{O}_2$ .  

$$\text{MnCl}_2 + (\text{NH}_4)_2\text{S} \rightleftharpoons \text{MnS} + 2\text{NH}_4\text{Cl}.$$
4. NaOH gives a white ppt. of  $\text{Mn}(\text{OH})_2$ ; insol. in excess, turning brown by oxidation, on standing, repptd. Ppt. not formed in presence of  $\text{NH}_4\text{Cl}$ .  

$$\text{MnCl}_2 + 2\text{NaOH} \rightleftharpoons \text{Mn}(\text{OH})_2 + 2\text{NaCl}.$$
  

$$2\text{Mn}(\text{OH})_2 + \text{O}_2 \rightarrow \text{Mn}_2\text{O}_3 + 2\text{H}_2\text{O}.$$

5.  $K_4FeCy_6$  gives a white ppt. of  $Mn_2FeCy_6$ .  
 $2MnCl_2 + K_4FeCy_6 = Mn_2FeCy_6 + 4KCl$ .

### Nickel, Ni.

1. HCl gives no ppt.
2.  $H_2S$  gives no ppt. in acid sol.
3.  $(NH_4)_2S$  gives a black ppt. of  $NiS$ ; sol. in excess of  $(NH_4)_2S$ , insol. in HCl, sol. in aqua regia.  
 $NiSO_4 + (NH_4)_2S = NiS + (NH_4)_2SO_4$ .
4. NaOH gives a light green ppt. of  $Ni(OH)_2$ ; insol. in excess.  
 $NiSO_4 + 2NaOH = Ni(OH)_2 + Na_2SO_4$ .
5.  $NH_4OH$  gives a similar ppt.; sol. in excess.
6. KCN gives a yellowish-green ppt. of  $NiCy_2$ ; sol. in excess.  
 $NiSO_4 + 2KCy = NiCy_2 + K_2SO_4$ .

### Cobalt, Co.

1. HCl gives no ppt.
2.  $H_2S$  gives no ppt. in acid sol.
3.  $(NH_4)_2S$  gives a black ppt. of  $CoS$ ; insol. in HCl, sol. in aqua regia.
4. NaOH gives a blue ppt. of  $Co(OH)_2 \cdot Co(NO_3)_2$ ; insol. in excess, becoming a rose-coloured ppt. of  $Co(OH)_2$  on heating.  
 $2Co(NO_3)_2 + 2NaOH = Co(OH)_2 \cdot Co(NO_3)_2 + 2NaNO_3$ .
5. KCN gives a brown ppt. of  $Co(Cy)_2$ ; sol. in excess.  
 $Co(NO_3)_2 + 2KCN = Co(CN)_2 + K_2SO_4$ ,  
 $Co(CN)_2 + 2KCN = 2KCN \cdot Co(CN)_2$ .

### Barium, Ba.

1. HCl,  $H_2S$ ,  $NH_4Cl + NH_4OH$  and  $(NH_4)_2S$  give no ppts..
2.  $(NH_4)_2CO_3$  in presence of  $NH_4Cl + NH_4OH$  gives a white ppt. of  $BaCO_3$ ; sol. in acids.  
 $BaCl_2 + (NH_4)_2CO_3 = BaCO_3 + 2NH_4Cl$ .  
 $BaCO_3 + 2HCl = BaCl_2 + H_2O + CO_2$ .
3.  $CaSO_4$  gives a heavy white ppt. of  $BaSO_4$ ; insol. in acids.  
 $BaCl_2 + CaSO_4 = BaSO_4 + CaCl_2$ .

4.  $\text{K}_2\text{CrO}_4$  gives a yellow ppt. of  $\text{BaCrO}_4$ ; almost insol. in  $\text{C}_2\text{H}_4\text{O}_2$ , sol. in HCl.  
 $\text{BaCl}_2 + \text{K}_2\text{CrO}_4 = \text{BaCrO}_4 + 2\text{KCl}$ .
5.  $\text{H}_2\text{SO}_4$  gives a white ppt. of  $\text{BaSO}_4$ .
6. Barium salts burn with a green-coloured flame.

**Strontrium, Sr.**

1. HCl,  $\text{H}_2\text{S}$ ,  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{OH}$  and  $(\text{NH}_4)_2\text{S}$  give no ppts.
2.  $(\text{NH}_4)_2\text{CO}_3$  in presence of  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$  gives a white ppt. of  $\text{SrCO}_3$ ; sol. in HCl.  
 $\text{SrCl}_2 + (\text{NH}_4)_2\text{CO}_3 = \text{SrCO}_3 + 2\text{NH}_4\text{Cl}$ .
3.  $\text{CaSO}_4$  gives a white ppt. of  $\text{SrSO}_4$  with difficulty or after agitation or warming.  
 $\text{SrCl}_2 + \text{CaSO}_4 = \text{SrSO}_4 + \text{CaCl}_2$ .
4.  $\text{H}_2\text{SO}_4$  also gives a white ppt. of  $\text{SrSO}_4$ .
5. Strontium salts burn with a crimson-coloured flame.

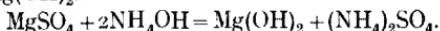
**Calcium, Ca.**

1. HCl,  $\text{H}_2\text{S}$ ,  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$  and  $(\text{NH}_4)_2\text{S}$  give no ppts.
2.  $(\text{NH}_4)_2\text{CO}_3$  in presence of  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$  gives a white ppt. of  $\text{CaCO}_3$ ; sol. in acids.  
 $\text{CaCl}_2 + (\text{NH}_4)_2\text{CO}_3 = \text{CaCO}_3 + 2\text{NH}_4\text{Cl}$ .
3.  $\text{H}_2\text{SO}_4$  gives a white ppt. of  $\text{CaSO}_4$ ; insol. in acids.  
 $\text{CaCl}_2 + \text{H}_2\text{SO}_4 = \text{CaSO}_4 + 2\text{HCl}$ .
4.  $\text{CaSO}_4$  gives no ppt.
5.  $(\text{NH}_4)_2\text{CO}_3$  gives a white ppt. of  $\text{CaCO}_3$ ; insol. in  $\text{C}_2\text{H}_4\text{O}_2$ , sol. in HCl.  
 $\text{CaCl}_2 + (\text{NH}_4)_2\text{CO}_3 = \text{CaCO}_3 + 2\text{NH}_4\text{Cl}$ .
6. Calcium salts burn with a brick-red coloured flame.

**Magnesium, Mg.**

1. HCl,  $\text{H}_2\text{S}$ ,  $\text{NH}_4\text{OH}$  and  $\text{NH}_4\text{Cl}$  and  $(\text{NH}_4)_2\text{S}$  or  $(\text{NH}_4)_2\text{CO}_3$  give no ppts.
2.  $\text{Na}_2\text{HPO}_4$  in presence of  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$  gives a white crystalline ppt. of  $\text{MgNH}_4\text{PO}_4$ ; sol. in HCl.  
 $\text{MgSO}_4 + \text{NH}_4\text{OH} + \text{Na}_2\text{HPO}_4 = \text{MgNH}_4\text{PO}_4 + \text{Na}_2\text{SO}_4 + \text{H}_2\text{O}$ .

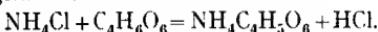
3.  $\text{NH}_4\text{OH}$ , in the absence of chlorides, gives a white ppt. of  $\text{Mg}(\text{OH})_2$ .



### Ammonium, $\text{NH}_3$ .

1.  $\text{HCl}$ ,  $\text{H}_2\text{S}$ ,  $\text{NH}_4\text{OH}$ ,  $\text{NH}_4\text{Cl}$  and  $(\text{NH}_4)_2\text{S}$  or  $(\text{NH}_4)_2\text{CO}_3$  or  $\text{Na}_2\text{HPO}_4$  give no ppts.

2. Tartaric acid gives a white ppt. in strong solutions with agitation.

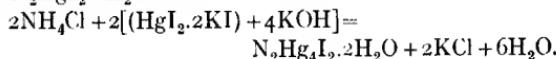


3. Boiled with  $\text{KOH}$  or  $\text{NaOH}$  evolves  $\text{NH}_3$ .

4.  $\text{PtCl}_4$  in  $\text{HCl}$  sols. gives a yellow crystalline ppt. of  $\text{PtCl}_4 \cdot 2\text{NH}_4\text{Cl}$ ; insol. in alcohol.



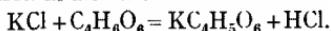
5. Nessler's solution gives a yellow to brown solution or ppt. of  $\text{N}_2\text{HgI}_2 \cdot 2\text{H}_2\text{O}$ .



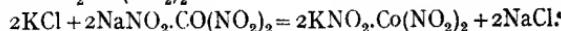
### Potassium, K.

1.  $\text{HCl}$ ,  $\text{H}_2\text{S}$ ,  $\text{NH}_4\text{OH}$ ,  $\text{NH}_4\text{Cl}$  and  $(\text{NH}_4)_2\text{S}$  or  $(\text{NH}_4)_2\text{CO}_3 \cdot \text{Na}_2\text{HPO}_4$  give no ppts.

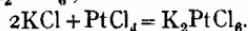
2. Tartaric acid in excess gives a white ppt. of  $\text{KC}_4\text{H}_5\text{O}_6$ ; insol. in alcohol.



3. *De Koninch's Test.*— $2\text{NaNO}_2 \cdot \text{Co}(\text{NO}_2)_2$  gives a yellow ppt. of  $2\text{KNO}_2 \cdot \text{CO}(\text{NO}_2)_2$ .



4. Platinum chloride in dilute solutions gives a yellow ppt. of  $\text{K}_2\text{PtCl}_6$ ; insol. in alcohol.



5. Potassium salts burn with a violet-coloured flame, best observed through a blue glass.

### Sodium, Na.

1.  $\text{HCl}$ ,  $\text{H}_2\text{S}$ ,  $\text{NH}_4\text{OH}$ ,  $\text{NH}_4\text{Cl}$  and  $(\text{NH}_4)_2\text{S}$ ,  $(\text{NH}_4)_2\text{CO}_3$  or  $\text{Na}_2\text{HPO}_4$ ,  $\text{C}_4\text{H}_6\text{O}_6$ ,  $\text{PtCl}_4$ , *De Koninch's test reagent*, give no ppts.

2.  $\text{KSbO}_3$  gives in neutral solutions a white ppt. of  $\text{Na}_2\text{H}_2\text{Sb}_2\text{O}_7$ .  

$$2\text{KSbO}_3 + 2\text{NaCl} + \text{H}_2\text{O} = \text{Na}_2\text{H}_2\text{Sb}_2\text{O}_7 + 2\text{KCl}$$
3. Sodium salts burn with an intense yellow-coloured flame.

### Sulphuric Acid, $\text{H}_2\text{SO}_4$ .

1.  $\text{BaCl}_2$  or  $\text{Ba}(\text{NO}_3)_2$  gives a white ppt. of  $\text{BaSO}_4$ ; insol. in acids or  $\text{NH}_4\text{OH}$ .  

$$(\text{NH}_4)_2\text{SO}_4 + \text{BaCl}_2 = \text{BaSO}_4 + \text{NH}_4\text{Cl}$$
2. Sulphates, when reduced with  $\text{Na}_2\text{CO}_3$  on charcoal, yield sodium sulphide, which stains a silver coin black ( $\text{Ag}_2\text{S}$ ).

### Sulphurous Acid, $\text{H}_2\text{SO}_3$ .

1. Sulphites, when treated with  $\text{HCl}$ , evolve  $\text{SO}_2$ , which reduces  $\text{K}_2\text{CrO}_4$  and liberates I from  $\text{KIO}_3$ .  

$$\text{Na}_2\text{SO}_3 + \text{HCl} = 2\text{NaCl} + \text{H}_2\text{O} + \text{SO}_2$$
  

$$4\text{SO}_2 + \text{K}_2\text{CrO}_4 = \text{K}_2\text{SO}_3 + \text{Cr}_2(\text{SO}_4)_3$$
.  

$$5\text{SO}_3 + 4\text{H}_2\text{O} + 2\text{KIO}_3 = \text{KHSO}_4 + 3\text{H}_2\text{SO}_4 + \text{I}_2$$
.
2.  $\text{BaCl}_2$  gives a white ppt.; insol. in  $\text{HCl}$  or  $\text{HNO}_3$ , which may be oxidized by Cl into  $\text{Na}_2\text{SO}_4$ .  

$$\text{Na}_2\text{SO}_3 + \text{BaCl}_2 = \text{BaSO}_4 + 2\text{NaCl}$$
.

### Hyposulphurous Acid, $\text{H}_2\text{S}_2\text{O}_3$ .

1.  $\text{BaCl}_2$  gives a white ppt. sol. in  $\text{HCl}$ , with liberation of sulphur.  

$$\text{Na}_2\text{S}_2\text{O}_3 + \text{BaCl}_2 = \text{BaS}_2\text{O}_3 + 2\text{NaCl}$$
.
2.  $\text{AgNO}_3$  gives a white ppt. of  $\text{Ag}_2\text{S}_2\text{O}_3$ , which gradually darkens to  $\text{Ag}_2\text{S}$ .  

$$\text{Na}_2\text{S}_2\text{O}_3 + 2\text{AgNO}_3 = \text{Ag}_2\text{S}_2\text{O}_3 + 2\text{NaNO}_3$$
.  

$$\text{Ag}_2\text{S}_2\text{O}_3 + \text{H}_2\text{O} = \text{Ag}_2\text{S} + \text{H}_2\text{SO}_4$$
.
3. Solutions containing Iodine are decolorized.  

$$2\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 = \text{Na}_2\text{S}_4\text{O}_6 + 2\text{NaI}$$
.
4.  $\text{HCl}$  liberates sulphur and sulphur dioxide.  

$$\text{Na}_2\text{S}_2\text{O}_3 + 2\text{HCl} = 2\text{NaCl} + \text{H}_2\text{O} + \text{S} + \text{SO}_2$$
.

### Carbonic Acid, $\text{H}_2\text{CO}_3$ .

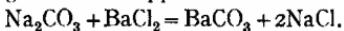
1. Dilute or strong acids liberate  $\text{CO}_2$ , with brisk effervescence, turning lime-water milky, forming calcium carbonate, which is sol. in excess of the gas.  

$$\text{Na}_2\text{CO}_3 + \text{HCl} = \text{CO}_2 + \text{H}_2\text{O} + 2\text{NaCl}$$
  

$$\text{CO}_2 + \text{Ca}(\text{OH})_2 = \text{CaCO}_3 + \text{HO}_2$$
.  

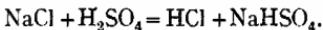
$$\text{CaCO}_3 + \text{CO}_2 + \text{H}_2\text{O} = \text{Ca}(\text{HCO}_3)_2$$
.

2.  $\text{BaCl}_2$  gives a white ppt. sol. in acids.

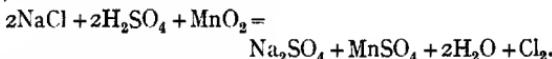


### Hydrochloric Acid, HCl.

1. Chlorides, when heated with conc.  $\text{H}_2\text{SO}_4$ , evolve HCl, which gives dense white fumes with  $\text{NH}_4\text{OH}$ !



2. Chlorides, when heated with conc.  $\text{H}_2\text{SO}_4$  and  $\text{MnO}_2$ , evolve Cl, which liberates I from KI.



3.  $\text{AgNO}_3$  gives a white ppt. insoluble in  $\text{HNO}_3$ , but sol. in  $\text{NH}_4\text{OH}$ .  $\text{KCN}$  or  $\text{Na}_2\text{S}_2\text{O}_3$ .

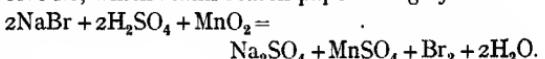


### Hydrobromic Acid, HBr.

1. Bromides, when heated with conc.  $\text{H}_2\text{SO}_4$ , evolve HBr (and Br).



2. Bromides, when heated with conc.  $\text{H}_2\text{SO}_4$  and  $\text{MnO}_2$ , evolve Br, which stains starch-paper orange-yellow.



3.  $\text{AgNO}_3$  gives a yellowish-white ppt.; slightly sol. in  $\text{NH}_4\text{OH}$ , insol. in dil.  $\text{HNO}_3$ .



### Hydriodic Acid, HI.

1. Iodides, when heated with conc.  $\text{H}_2\text{SO}_4$ , evolve I mixed with  $\text{SO}_2$ , which turns starch-paper blue.



2. Iodides heated with  $\text{H}_2\text{SO}_4$  and  $\text{MnO}_2$  evolve I.



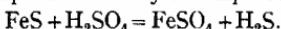
3.  $\text{AgNO}_3$  gives a yellow ppt. of  $\text{AgI}$ ; insol. in  $\text{NH}_4\text{OH}$  and in dil.  $\text{HNO}_3$ .



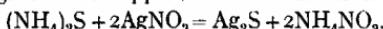
### Sulphuretted Hydrogen, $\text{H}_2\text{S}$ .

- x. Sulphides heated in air or oxygen evolve  $\text{SO}_2$ .

2. Sulphides, in general, evolve  $H_2S$  on treatment with  $HCl$  or  $H_2SO_4$ , which blackens lead acetate paper. Certain sulphides are only decomposed when heated with acids.



3.  $AgNO_3$  gives a black ppt. ; insol. in dil. acids.



### Nitric Acid, $HNO_3$ .

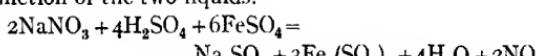
1. Nitrates, when heated with conc.  $H_2SO_4$ , evolve  $HNO_3$  and brownish-yellow vapours composed of the lower oxides of N.



2. Nitrates heated with  $H_2SO_4$  in presence of metallic copper evolve red fumes of  $NO_2$ .



3. Solutions of nitrates, when treated with  $H_2SO_4$ , and freshly prepared  $FeSO_4$ , in the cold, form a brown ring at the junction of the two liquids.



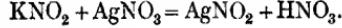
4. Brucine, in presence of  $H_2SO_4$ , gives a red ring.

### Nitrous Acid, $HNO_2$ .

1. Nitrites, when heated with dil. acids, evolve  $N_2O_2$ , which rapidly oxidizes in air, forming red fumes of  $N_2O_3$  and  $N_2O_4$ .

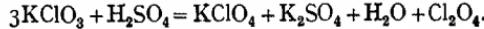


2.  $AgNO_3$  gives a white ppt. of  $AgNO_3$  ; sol. in excess of water.



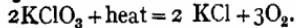
### Chloric Acid, $HClO_3$ .

1. Chlorates heated with  $H_2SO_4$  detonate, and evolve  $Cl_2O_4$ — a greenish-yellow, highly explosive gas.



2. Chlorates heated with carbon deflagrate.

3. Chlorates heated alone decrepitate, and evolve oxygen.



4. Indigo is bleached by solutions of chlorates slightly acidified with  $H_2SO_4$ .

**Chromic Acid,  $H_2CrO_4$ .**

1. Chromates heated with  $H_2SO_4$  and  $NaCl$  yield red fumes of chromyl-chloride.
2.  $BaCl_2$  gives a yellow ppt.; sol. in  $HCl$  and in  $HNO_3$ , insol. in  $C_2H_4O_2$ .  
 $K_2CrO_4 + BaCl_2 = BaCrO_4 + 2KCl$ .
3.  $Pb(NO_3)_2$  or  $Pb(C_2H_4O_2)_2$  gives a yellow ppt.; sol. in  $NaOH$ , insol. in  $C_2H_4O_2$ .
4.  $AgNO_3$  gives a dark red ppt.; sol. in  $HNO_3$  or  $NH_4OH$ .

**Arsenious Acid,  $H_3AsO_3$ .**

1.  $AgNO_3$  gives in neutral solutions a yellow ppt.; sol. in  $NH_4OH$ .  
 $3AgNO_3 + H_3AsO_3 = Ag_3AsO_3 + 3HNO_3$ .
2.  $MgSO_4$  in presence of  $NH_4Cl$  and  $NH_4OH$  gives no ppt.

**Arsenic Acid,  $H_3AsO_4$ .**

1.  $AgNO_3$  gives in neutral solutions a reddish-brown ppt.; sol. in  $HNO_3$ .
2.  $MgSO_4$  in presence of  $NH_4Cl$  and  $NH_4OH$  gives a white ppt. of  $MgNH_4AsO_4$ .

**Hydrofluoric Acid, HF.**

1. Fluorides treated with conc.  $H_2SO_4$  evolve HF, which etches glass.
 
$$CaF_2 + H_2SO_4 = CaSO_4 + 2HF$$

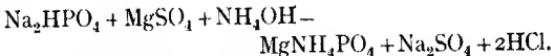
$$SiO_2 + 4HF = SiF_4 + 2HO$$

$$SiF_4 + 4H_2O = H_4SiO_4 + 2H_2SiF_6$$
2.  $BaCl_2$  gives a white ppt.; sol. in  $HCl$ .
 
$$2HF + BaCl_2 = BaF_2 + 2KCl$$
3.  $CaCl_2$  gives a gelatinous ppt. of  $CaF_2$ .

**Orthophosphoric Acid,  $H_3PO_4$ .**

1.  $BaCl_2$  gives a white ppt.; sol. in  $HCl$  or  $HNO_3$ .
2.  $CaCl_2$  gives a white ppt.; sol. in  $HCl$ ,  $HNO_3$ , or  $C_2H_4O_2$ .
 
$$Na_2HPO_4 + CaCl_2 = CaHPO_4 + 2NaCl$$
3.  $Fe_2Cl_6$  gives a yellowish-white ppt. of  $FePO_4$ ; sol. in  $HCl$  or  $HNO_3$ , insol. in  $C_2H_4O_2$ .
 
$$2Na_2HPO_4 + Fe_2Cl_6 = 2FePO_4 + 4NaCl + 2HCl$$

4.  $\text{MgSO}_4$  gives, in presence of  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$ , a white ppt.



5.  $\text{AgNO}_3$  gives a yellow ppt.; sol. in acids or  $\text{NH}_4\text{OH}$ .



6.  $(\text{NH}_4)_2\text{MoO}_4$ , in excess, in presence of conc.  $\text{HNO}_3$ , gives, on gently warming, a canary-yellow ppt. of  $2(\text{NH}_4)_2\text{PO}_4 \cdot 22\text{MoO}_4 \cdot 12\text{H}_2\text{O}$ ; insol. in acids, sol. in  $\text{NH}_4\text{OH}$ .

#### **Metaphosphoric Acid, $\text{HPO}_3$ .**

1.  $\text{AgNO}_3$  gives a white gelatinous ppt. of  $\text{AgNO}_3$ .

2.  $\text{MgSO}_4$ , in presence of  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$ , gives no ppt.

3. As free acid, it ppts. albumin.

#### **Pyrophosphoric Acid, $\text{H}_4\text{P}_2\text{O}_7$ .**

1.  $\text{AgNO}_3$  gives a white ppt. of  $\text{Ag}_4\text{P}_2\text{O}_7$ ; sol. in  $\text{HNO}_3$  or  $\text{NH}_4\text{OH}$ .

2.  $\text{MgSO}_4$  gives a white ppt. of  $\text{Mg}_2\text{P}_2\text{O}_7$ ; sol. in excess.

3. As free acid, it ppts. albumin.

#### **Boric Acid, $\text{H}_3\text{BO}_3$ .**

1. Borates moistened with  $\text{H}_2\text{SO}_4$  and alcohol burn with a green flame.

2.  $\text{BaCl}_2$  gives a white ppt.; sol. in acids.

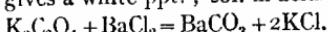
3.  $\text{AgNO}_3$  gives a white ppt. in concentrated solutions.

#### **Oxalic Acid, $\text{H}_2\text{C}_2\text{O}_4$ .**

1. Oxalates heated with conc.  $\text{H}_2\text{SO}_4$  do not char, but evolve  $\text{CO}$  and  $\text{CO}_2$ .



2.  $\text{BaCl}_2$  gives a white ppt.; sol. in acids.



3.  $\text{CaCl}_2$  gives a white ppt.; sol. in  $\text{HCl}$  or  $\text{HNO}_3$ , but insol. in  $\text{C}_2\text{H}_4\text{O}_2$  or  $\text{NH}_4\text{OH}$ .

#### **Tartaric Acid, $\text{C}_4\text{H}_6\text{O}_6$ .**

1. Tartrates heated alone in a dry tube char and give off caramel odour.

2. Tartrates, when heated with conc.  $H_2SO_4$ , char rapidly, and evolve CO and  $CO_2$ .
3.  $CaCl_2$ , in neutral solutions, gives a white ppt.; sol. in acids and in conc. cold KOH repprd. on boiling.  

$$CaCl_2 + KNaC_4H_4O_6 = CaC_4H_4O_6 + KCl + NaCl.$$
4.  $AgNO_3$  gives a white ppt.; sol. in  $HNO_3$  and  $NH_4OH$ . The ppt. dissolved in the minimum quantity of  $NH_4OH$  deposits on heating the Ag as a metallic mirror.  

$$2AgNO_3 + KNaC_4H_4O_6 = Ag_2C_4H_4O_6 + KNO_3 + NaNO_3.$$

**Citric Acid,  $C_6H_8O_7$ .**

1. Citrates, heated alone in a dry tube, char, and evolve irritating fumcs.
2. Citrates heated with conc.  $H_2SO_4$  char slowly, evolvng CO, and subsequently  $CO_2$ ,  $SO_2$ , etc.
3.  $CaCl_2$  gives, on boiling in neutral solutions, a white ppt. (no ppt. in the cold); insol. in KOH, sol. in  $NH_4OH$ .  

$$3CaCl_2 + 2K_3C_6H_5O_7 = 6KCl + Ca_3(C_6H_5O_7)_2.$$
4.  $AgNO_3$  gives a white ppt.; sol. in  $NH_4OH$ , which deposits the Ag, on prolonged boiling, as a greyish amorphous ppt.  

$$3AgNO_3 + K_3C_6H_5O_7 = 6KNO_3 + Ag_3C_6H_5O_7.$$

**Benzoic Acid,  $C_7H_6O_2$ .**

1. Benzoates heated alone in a dry tube sublime and evolve an irritating odour; when mixed with CaO benzene is given off. Heated on pt. foil, benzoates ignite, burning with a smoky flame.  

$$CaO + C_7H_6O_2 = CaCO_3 + C_6H_6.$$
2. Benzoates do not char when treated with conc.  $H_2SO_4$ , benzoic acid being evolved.
3.  $Fe_2Cl_6$  gives a buff-coloured ppt. in neutral solutions; sol. in HCl.  

$$6NH_4C_7H_5O_2 + 2Fe_2Cl_6 = Fe_2(C_7H_5O_2)_6 + 6NaCl.$$
  

$$Fe_2(C_7H_5O_2)_6 + 6HCl = Fe_2Cl_6 + 6C_7H_6O_2.$$

**Formic Acid,  $CH_2O_2$ .**

1. Formates, heated with conc.  $H_2SO_4$ , evolve CO without charring.  

$$KHCO_3 + H_2SO_4 = KHSO_4 + H_2O + CO.$$

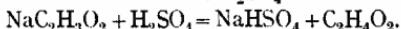
2.  $\text{AgNO}_3$ , in neutral solutions, gives a white ppt., which rapidly darkens as the metallic Ag is deposited.



3.  $\text{HgCl}_2$  and  $\text{Hg}(\text{NO}_3)_2$  both give white ppts., turning to grey metallic Hg.

### Acetic Acid, $\text{C}_2\text{H}_4\text{O}_2$ .

1. Acetates heated with conc.  $\text{H}_2\text{SO}_4$  evolve acetic acid.



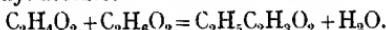
2.  $\text{AgNO}_3$  gives a white ppt. in conc. solutions; sol. in  $\text{NH}_4\text{OH}$ .



3.  $\text{Fe}_2\text{Cl}_6$  gives a red coloration, which becomes a brown ppt. on boiling.

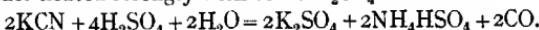
4. Acetates do not reduce silver or mercury salts.

5. Acetates heated with conc.  $\text{H}_2\text{SO}_4$  and ethyl alcohol evolve ethyl acetate.



### Hydrocyanic Acid, HCN.

1. Cyanides heated strongly with conc.  $\text{H}_2\text{SO}_4$  evolve CO.

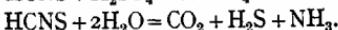
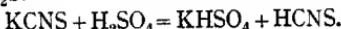


2. Cyanides treated with HCl evolve HCN gas, which may be converted into sulphocyanide by treatment with  $(\text{NH}_4)_2\text{S}$ , yielding a blood-red coloration on the addition of a few drops of HCl and  $\text{Fe}_2\text{Cl}_6$ .

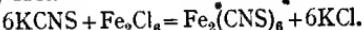
3.  $\text{AgNO}_3$  gives a white ppt.; insol. in  $\text{HNO}_3$ , sol. in  $\text{NH}_4\text{OH}$  or KCN.

### Sulphocyanic Acid, HCNS.

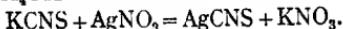
1. Sulphocyanides heated with  $\text{H}_2\text{SO}_4$  evolve HCN,  $\text{CO}_2$ , and  $\text{H}_2\text{S}$ .



2.  $\text{Fe}_2\text{Cl}_6$  gives a blood-red ppt.; destroyed by  $\text{HgCl}_2$ , but not by HCl.

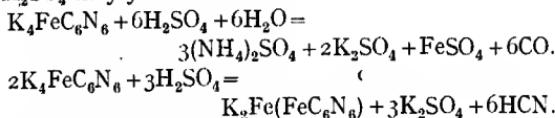


3.  $\text{AgNO}_3$  gives a white ppt.; insol. in dil.  $\text{HNO}_3$ , sol. in  $\text{NH}_4\text{OH}$  and KCNS.

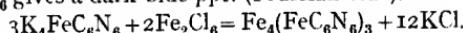


**Ferrocyanic Acid,  $H_4FeC_6N_6$ .**

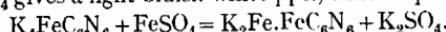
1. Ferrocyanides heated with conc.  $H_2SO_4$  evolve CO; with dil.  $H_2SO_4$  they yield HCN.



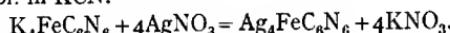
2.  $Fe_2Cl_6$  gives a dark blue ppt. (Prussian blue).



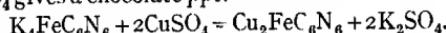
3.  $FeSO_4$  gives a light bluish-white ppt., which rapidly darkens.



4.  $AgNO_3$  gives a white ppt.; insol. in dil.  $HNO_3$  or  $NH_4OH$ , sol. in  $KCN$ .



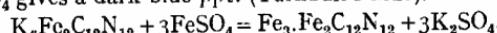
5.  $CuSO_4$  gives a chocolate ppt.

**Ferricyanic Acid,  $H_6Fe_2C_{12}N_{12}$ .**

1. Ferricyanides heated with conc.  $H_2SO_4$  evolve CO and  $CO_2$ .

2.  $Fe_2Cl_6$  gives a dark greenish coloration.

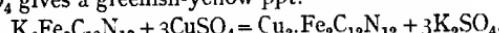
3.  $FeSO_4$  gives a dark blue ppt. (Turnbull's blue).



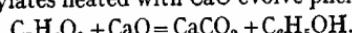
4.  $AgNO_3$  gives an orange ppt.; insol. in dil.  $HNO_3$ , sol. in  $KCN$ .



5.  $CuSO_4$  gives a greenish-yellow ppt.

**Salicylic Acid,  $C_7H_6O_3$ .**

1. Salicylates heated with  $CaO$  evolve phenol.



2. Warmed with  $H_2SO_4$  and methyl alcohol, salicylates evolve methyl salicylate.

3.  $Fe_2Cl_6$  gives an intense violet coloration, destroyed by acids and alkalies.

4. Bromine-water gives a yellowish-white ppt.

**Carbolic Acid,  $C_6H_6OH$ .**

1. Carbolates heated alone or with  $H_2SO_4$  evolve phenol (carbolic acid).

2.  $Fe_2Cl_6$  gives a reddish-violet coloration.

3. NaClO gives in a slightly ammoniacal solution a deep blue coloration, turning red on acidifying.
4. Bromine-water gives a yellowish-white ppt. of tribromo-phenol ( $C_6H_2Br_3OH$ ).

**Tannic Acid,  $C_{27}H_{22}O_{17}$ .**

1. Tannates, when heated, char and evolve a caramel odour ; when heated with  $H_2SO_4$  they yield a dark green liquid with irritating fumes.
2.  $Fe_2Cl_6$  gives a greenish or bluish-black ppt. (ink).
3. Solutions of gelatine give a greyish ppt. even in dilute solution.

**Uric Acid,  $C_5H_4N_1O_3$ .**

1. Urates char immediately on heating, and evolve an odour of burnt feathers ; heated with  $H_2SO_4$ , they yield  $CO_2$  and  $SO_2$ .
2. Dissolved in  $HNO_3$  and evaporated to dryness, a bright purple coloration is produced when the residue is drenched with  $NH_4OH$ .

**Urea,  $C_4H_8N_2O$ .**

1. Urea is sol. in water or alcohol, insol. in ether.
2. Heated to  $150^\circ C.$ , ammonia is evolved
3.  $HNO_3$  liberates  $H_2O$ ,  $CO_2$ , and N.
4. Alkalies on heating evolve  $NH_3$ .

**Morphine,  $C_{17}H_{19}NO_3$ .**

1. Morphine is insol. in water, sol. in alcohol or acids.
2.  $Fe_2Cl_6$  in neutral solutions gives a dark blue colour, changing to green on adding excess of the reagent, destroyed by HCl.
3.  $HNO_3$ , strong and in excess, produces an orange coloration.
4. Alkalies ppt. morphine from its solutions ; sol. in excess of the reagent.
5. If a little  $H_2SO_4$  be added to a solution containing morphine and the resultant colourless liquid be divided into two parts, (1)  $HNO_3$  will yield a blood-red coloration ; (2) a crystal of  $K_2Cr_2O_7$ , a green coloration.

**Quinine,  $C_{26}H_{24}N_2O$ .**

1. Slightly sol. in water, sol. in alcohol, chloroform, and most acids.

2. KOH gives a white ppt. of the alkaloid ; insol. in excess, sol. in ether.
3. Chlorine-water, to an ac'd solution, gives a green coloration on addition of  $\text{NH}_4\text{OH}$ .

**Cinchonine**,  $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}$ .

1. Less soluble than quinine.
2. KOH gives a white ppt. of the alkaloid ; insol. in excess or in ether.
3. Chlorine-water gives no coloration, but, on addition of  $\text{NH}_4\text{OH}$ , a yellowish ppt.

**Brucine**,  $\text{C}_{23}\text{H}_{26}\text{N}_2\text{O}_4$ .

1. Slightly soluble in water ; sol. in alcohol, chloroform, and most acids.
2.  $\text{HNO}_3$  gives a pink colour, turning yellow on heating, and purple on the addition of  $\text{SnCl}_2$ .
3.  $\text{HNO}_3$  in solution, containing a layer of  $\text{H}_2\text{SO}_4$ , gives a red ring at the junction of the two liquids.
4.  $\text{H}_2\text{SO}_4$  gives a fugitive rose-red coloration.

**Strychnine**,  $\text{C}_{21}\text{H}_{23}\text{N}_2\text{O}_2$ .

1. Slightly soluble in water, alcohol, and ether ; sol. in chloroform and in most acids ; sol. in  $\text{H}_2\text{SO}_4$ , without change in colour, yielding in this solution the following protean colorations :
  - (a)  $\text{MnO}_2$ —a violet to crimson, and finally orange.
  - (b)  $\text{K}_2\text{CrO}_4$ —a blue to reddish-yellow.
2.  $\text{HNO}_3$ ; no change in the cold, yellow when heated.

**Nicotine**,  $\text{C}_{10}\text{H}_{14}\text{N}_2$ .

1. A colourless oily liquid, oxidizing to a brown colour ; sol. in water, alcohol, and ether with alkaline reaction.
2. When warmed, yields dense white fumes in presence of conc. HCl.
3. A solution of I and KI gives a transient yellow ppt.
4.  $\text{HNO}_3$  gives no red coloration.

**Albumin.**

1. Albumin is coagulated on heating, forming an insoluble mass, and is ptd. more or less by most acids and certain salts (in particular,  $\text{HgCl}_2$ ).

**Methyl Alcohol,  $\text{CH}_3\text{OH}$ .**

1. When heated alone, it evaporates without change.
2. Chars with  $\text{H}_2\text{SO}_4$ .
3. Free iodine gives no iodofrom.
4. Oxidizes to formic acid.

**Ethyl Alcohol,  $\text{C}_2\text{H}_5\text{OH}$ .**

1. When heated alone, it evaporates without change.
2. Does not char with  $\text{H}_2\text{SO}_4$ , but yields  $(\text{C}_2\text{H}_5)_2\text{O}$  or  $\text{C}_2\text{H}_4$ .
3. Heated with KOH and I + KI, a yellow ppt. of iodofrom is produced.
4. Oxidizes to acetic acid.

**Glycerine,  $\text{C}_2\text{H}_5(\text{OH})_3$ .**

1. Heated alone, chars and evolves irritating fumes of acrolein.

**Sucrose,  $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .**

1. Chars when treated with conc.  $\text{H}_2\text{SO}_4$  in the cold, yielding CO and  $\text{SO}_2$  on heating.
2. Does not reduce Fehling's solution until inverted by dilute acids.

**Dextrose,  $\text{C}_6\text{H}_{12}\text{O}_6$ .**

1. Does not char in the cold with conc.  $\text{H}_2\text{SO}_4$ .
2. Reduces Fehling's solution, on heating, to a red ppt. ( $\text{Cu}_2\text{O}$ ).

**Maltose,  $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .**

1. Heated with conc.  $\text{H}_2\text{SO}_4$ , chars slowly.
2. Reduces Fehling's solution, on heating, to a red ppt. ( $\text{Cu}_2\text{O}$ ).

**Lactose,  $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .**

1. Slowly charred in the cold by conc.  $\text{H}_2\text{SO}_4$ .
2. Reduces Fehling's solution, on heating, to a red ppt. ( $\text{Cu}_2\text{O}$ ).

**Cellulose,  $\text{C}_6\text{H}_{10}\text{O}_5$ .**

1. Insoluble in water or alcohol.
2. Is not turned blue in presence of free iodine.

**Starch,  $\text{C}_6\text{H}_{10}\text{O}_5$ .**

1. Insoluble in water, forming a paste.
2. Turned blue in presence of free iodine.
3. Converted at  $150^\circ \text{C}.$ , or on gently warming with dil. acids or enzymes, into dextrin.

**Dextrin,  $\text{C}_6\text{H}_{10}\text{O}_5$ .**

1. Soluble in water, forming a gum.
2. Is not turned blue in presence of free iodine.
3. Is converted by dil. acids into sugars.

### Table for the Separation of the Common Metals.

- I. Silver Group.**—Add HCl; white ppt. = PbCl<sub>2</sub>, AgCl, Hg<sub>2</sub>Cl<sub>2</sub>. Filter and wash with cold water.  
**Pb', Ag', Hg'.** Pour boiling water on filter. PbCl<sub>2</sub> dissolves; test part of filtrate with K<sub>2</sub>CrO<sub>4</sub>, and the rest with KI : yellow ppts. = Pb.  
**Ag, Hg'.** Pour warm NH<sub>4</sub>OH on filter. AgCl dissolves ; test part of filtrate with HNO<sub>3</sub>; AgCl reppd.; test the rest with K<sub>2</sub>CrO<sub>4</sub>; red ppt. = Ag.  
**Hg'.** If ppt. blackens, Hg is present. Dissolve the ppt. in aqua regia. Boil down to small bulk, and add SnCl<sub>2</sub>; white ppt. turning to grey = Hg.
- II. Copper-Arsenic Group.**—Warm filtrate from Group I., and pass H<sub>2</sub>S; a black ppt. = HgS, PbS, Bi<sub>2</sub>S<sub>3</sub>, CuS, SnS ; yellow ppt. = CdS, As<sub>2</sub>S<sub>3</sub>, SnS<sub>2</sub>; orange ppt. = Sb<sub>2</sub>S<sub>3</sub>. Wash ppt. free from HCl, and digest it for some time with NaOH or yellow (NH<sub>4</sub>)<sub>2</sub>S. Dilute with water, and filter. Residue = Hg. Confirm as in Group I.: grey ppt. = Hg.
- A. Residue = Pb'', Bi, Cu, Cd. Hg''.** Pour on boiling dilute HNO<sub>3</sub>. Add H<sub>2</sub>SO<sub>4</sub> to part of filtrate; white ppt. = Pb. Neutralize and confirm by K<sub>2</sub>CrO<sub>4</sub>; yellow ppt. = Pb.
- Pb'', Bi, Cu, Cd.** To filtrate from Pb, add NH<sub>4</sub>OH : white ppt. = Bi. Filter, dissolve ppt. in a few drops HCl, add a little water; milkiness = Bi.
- Cu, Cd.** If filtrate is blue = Cu.  
**Cd.** To rest of filtrate add KCN ; pass H<sub>2</sub>S : yellow ppt. = Cd.
- B. Filtrate = As, Sn, Sb.** Reptt. by adding HCl ; wash, digest with solid (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>; As<sub>2</sub>S<sub>3</sub> dissolves. Filter and reptt. with HCl ; yellow ppt. = As.
- Sn, Sb.** Dissolve residue in conc. HCl. Place Pt and Zn foil in solution : Sn is deposited on Zn. Dissolve in HCl, and add HgCl<sub>2</sub>; white to grey ppt. = Sn.  
**Sb.** Is deposited on Pt. Dissolve in warm HNO<sub>3</sub>, and pass H<sub>2</sub>S; orange ppt. = Sb.

**III. Iron Group.**—(A) Boil filtrate from Group II, till free from  $H_2S$ , and oxidize with  $HNO_3$ . Test for  $H_3PO_4$ , and add  $NH_4OH + NH_4Cl$ . If ppt. brown = Fe; white = Al.  
 N.B.—If  $H_3PO_4$  is present,  $NH_4OH$  gives a white ppt.; also with Ca and Mg groups, which is soluble in acetic acid.

(B) To filtrate add  $(NH_4)_2S$ . If ppt. is flesh-coloured = Mn; white = Zn; black = Co and Ni. Filter, wash with water containing  $(NH_4)_2S$ , and then pure water. Residue = Fe.

A = Fe, Al, Cr. Fuse with  $Na_2CO_3 + KNO_3$ , and boil with water. Residue = Fe.  
 Filter. Dissolve in  $HCl$ , and test with  $K_3Fe(CN)_6$ ; Prussian blue = Fe.  
 Al, Cr. To filtrate add  $NH_4Cl$ , and boil. White ppt. = Al; add acetic acid till acid and lead acetate; yellow ppt. = Cr.  
 B = Mn, Zn, Co, Ni. Pour cold dilute  $HCl$  on ppt.;  $MnS$  and  $ZnS$  dissolve = (a).  
 $CoS$  remain undissolved = (b).  
 (a) Mn, Zn. Boil till  $H_2S$  free; cool; add  $NaOH$ ; white ppt. turning to brown = Mn. To filtrate from Mn, add  $(NH_4)_2S$ ; white ppt. = Zn.  
 (b) Co, Ni. Test for Co by borax bead = blue. For Ni dissolve in  $HCl + KClO_3$ ; neutralize with  $NH_4OH$ ; add  $KCN$  till ppt. first formed is dissolved; add acetic acid and  $NaClO$ , and warm; black ppt. = Ni.

**IV. Barium Group.**—Add  $(NH_4)_2CO_3$ . White ppt. =  $BaCO_3$ ,  $CaCO_3$ , or  $S_2CO_3$ .

Dissolve ppt. in acetic acid :

- (1) To part add  $K_2CrO_4$ ; yellow ppt. = Ba. To filtrate from Ba. add  $CaSO_4$ , and boil; white ppt. = Sr.
- (2) To rest add  $H_2SO_4$ , and boil; filter; add  $NH_4OH$  and  $(NH_4)_2C_2O_4$ ; white ppt. = Ca.

#### •V. Potassium Group.

Mg. Concentrate part of solution, and add  $Na_2HPO_4$ ; white ppt. = Mg.  
 K. Take another part of filtrate from Ba group; evaporate to dryness, ignite residue, dissolve in water, add  $HCl + PtCl_4$  + alcohol; yellow ppt. = K.  
 Na. Test another portion by flame; yellow flame = Na.  
 $NH_4$ . Test by boiling with  $NaOH$  in original solution (preliminary tests).

**Table of Common Inorganic Acids.**

$\text{H}_2\text{CO}_3$ ,  $\text{H}_2\text{S}$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{SO}_3$ ,  $\text{HCl}$ ,  $\text{HBr}$ ,  $\text{HI}$ ,  $\text{HF}$ ,  $\text{H}_3\text{PO}_4$ ,  $\text{HNO}_3$ ,  
 $\text{HClO}_3$ .

A. Add dilute  $\text{HCl}$ ; warm, and observe if effervescence; if so, may be—

- (a)  $\text{H}_2\text{CO}_3$ .— $\text{CO}_2$ : gives white ppt. with lime-water.
- (b)  $\text{H}_2\text{S}$ .— $\text{H}_2\text{S}$ : gives black ppt. with lead acetate.
- (c)  $\text{H}_2\text{SO}_3$ .— $\text{SO}_2$ : smell of burning sulphur and bleaching  $\text{KMnO}_4$  solution.

N.B.—If a chlorate is present, chlorine is here given off.

B. Heat with conc.  $\text{H}_2\text{SO}_4$ , when, in addition to "A," acid fumes may be evolved— $\text{HCl}$  from chlorides,  $\text{HBr}$  and  $\text{Br}$  from bromides,  $\text{HI}$  and  $\text{I}$  from iodides,  $\text{Cl}_2\text{O}_4$  with explosions from chlorates,  $\text{HNO}_3$  from nitrates,  $\text{HF}$  from fluorides. Confirm for  $\text{HF}$  by etching glass, for  $\text{HNO}_3$  by "ring" test, copper test, and indigo test. The latter is also appreciable if  $\text{HClO}_3$  is present.

C. Heat another portion with  $\text{H}_2\text{SO}_4$  and  $\text{MnO}_2$ , when—

Cl given off, recognised by its odour and action on KI and starch.

I given off, recognised by its violet vapours and its action on starch paste.

Br given off, recognised by its reddish-brown fumes and smell, etc.

D. If bases other than the alkalies are present, boil a portion of the original solution or substance with a slight excess of  $\text{Na}_2\text{CO}_3$ . Filter, divide filtrate into two parts :

1. Boil with  $\text{HCl}$  till effervescence ceases; add  $\text{BaCl}_2$ .  
a white ppt. =  $\text{H}_2\text{SO}_4$ .
2. Boil with  $\text{HNO}_3$  till effervescence ceases; add  $\text{AgNO}_3$ .
  - (a) White ppt., easily sol. in  $\text{NH}_4\text{OH}$ . =  $\text{HCl}$ .
  - (b) Yellowish-white ppt., difficultly sol. in  $\text{NH}_4\text{OH}$ . =  $\text{HBr}$ .
  - (c) Yellow ppt., nearly insol. in  $\text{NH}_4\text{OH}$ . =  $\text{HI}$ .

N.B.—Separate  $\text{HBr}$ ,  $\text{HI}$ , by  $\text{CS}_2$  test, and  $\text{HCl}$  from these by distilling with  $\text{K}_2\text{CrO}_4$  and  $\text{H}_2\text{SO}_4$ .

E. After removal of copper and arsenic groups, if these be present, test for  $\text{H}_3\text{PO}_4$  by adding  $\text{HNO}_3$  and ammonium molybdate—yellow ppt. =  $\text{H}_3\text{PO}_4$ .

**Table for the Separation of the Common Organic Acids.**

1. Remove inorganic bases, if present, by boiling with  $\text{Na}_2\text{CO}_3$ ; filter; acidify with  $\text{HNO}_3$ , and expel  $\text{CO}_2$  by warming; neutralize with  $\text{NaOH}$ .  
 2. In the absence of bases, add  $\text{Na}_2\text{CO}_3$  in excess; boil; add  $\text{HNO}_3$  till exactly neutral. Add  $\text{CaCl}_2$  to the neutral solution, and allow to stand for ten minutes; filter.

<b>RESIDUE.</b> May contain oxalic and tartaric acids. Wash well; add conc. cold KOH. Filter.	<b>FILTRATE.</b> May contain the other acids. Boil and filter.		
<b>RESIDUE.</b> Citrate; sol. in $\text{HCl}$ , but repprd. by $\text{NH}_4\text{OH}$ and on boiling.	<b>FILTRATE.</b> Add three volumes of alcohol. Filter.	<b>RESIDUE.</b> Succinate and malate. Dissolve in $\text{HNO}_3$ , and evaporate nearly to dryness; add excess $\text{Na}_2\text{CO}_3$ . Boil; filter; neutralize with $\text{HCl}$ ; divide into two portions—A and B.	<b>FILTRATE.</b> Evaporate off the alcohol, and neutralize with $\text{HCl}$ ; add $\text{Fe}_2\text{Cl}_9$ . Buff ppt. = benzoic. Filter.
<b>RESIDUE.</b> Filtrate. Tartrate repprd. on boiling.		<b>RESIDUE.</b> Add $\text{CaSO}_4$ . Add $\text{CaCl}_2$ in excess, and filter; White ppt. = malic.	<b>RESIDUE.</b> May contain formic, acetic, etc.  <b>RESIDUE.</b> Benzoic.  <b>RESIDUE.</b> Gelatinous ppt. = succinic.
			<b>FILTRATE.</b>

## CHAPTER III

### GRAVIMETRIC ESTIMATIONS

#### Estimation of Silica.

**Insoluble Silica.**—The silica is rendered insoluble and alone by treatment with excess of acid, the whole is evaporated to dryness, taken up with a little more acid, filtered, washed, and dried, or ignited without previously being dried, and weighed as silica ( $\text{SiO}_2$ ).

**Soluble Silicate decomposed by Strong Acids.**—Weigh out into a platinum capsule 1 gramme silicate of soda, moisten it with a little water, and heat over a water-bath, with frequent addition of strong hydrochloric acid together with stirring, until all is dissolved. Evaporate the solution to dryness, drench with strong hydrochloric acid, dilute with hot water, and decant off the supernatant liquid through a filter. Wash the residue in this way three times with hot water, pouring the washings each time through the filter. Lastly, transfer the residue of silica on to the filter, wash thoroughly with hot water until the wash water is no longer acid, dry the precipitate in the water-oven. Ignite the filter, and then add the ash to the dry silica, heat strongly, cool, and weigh as  $\text{SiO}_2$ .

The ignited powder should be pure white in colour in every case where any degree of accuracy is required. The residue still retained in the platinum capsule should be heated over the water-bath with pure hydrofluoric acid and a little strong sulphuric acid, evaporated to dryness, ignited, and again weighed, the treatment with hydrofluoric acid being repeated until no further loss is observed.

The total loss thus produced equals the amount of silica present.

**Partially Soluble Silicate.**—The substance must be very finely ground in an agate mortar, so that no grit is felt when it is

bit with the teeth. A weighed quantity is fused in a platinum crucible with at least six times its weight of fusion mixture ( $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{CO}_3$ ) for about twenty minutes, or at least until the molten mass becomes quite tranquil. The hot crucible is then half immersed in cold water contained in a porcelain evaporating basin, and is at the same time held steady by the tongs. In this way the melt is detached from the crucible more easily than by boiling it wholly immersed in water.

The carbonates are dissolved by the addition of a little hydrochloric acid, and the whole liquid is evaporated to dryness, the residue ignited, and then proceeded with as in an insoluble silicate for  $\text{SiO}_2$ .

#### Estimation of Water, Free and Combined.

**Moisture.**—Substances which give up their moisture on heating up to 100° C., without otherwise changing in their chemical state, may be placed in shallow porcelain dishes or tared clipped watch-glasses, and subjected to the heat of the water-oven for a period of two or more hours; then cooled, weighed, and again heated until no further loss in weight is observed.

**Combined Water.**—1. Substances which require a higher temperature than 100° C. in order to lose their water, and which are not altered otherwise at such temperatures, may be heated in a suitable dish or crucible over a Bunsen burner for twenty minutes, cooled, weighed, and again reheated until a constant weight is obtained.

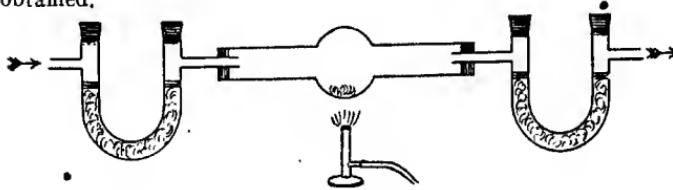


FIG. 14.—U-TUBES.

2. Substances which are altered by being strongly heated may be placed in a bulbed ignition tube of Jena glass (as shown), connected at each end with tared U-tubes containing calcic chloride, the limbs of which are plugged with cotton-wool and securely sealed.

The side tubes are fitted with short pieces of rubber tubing into which glass rods are inserted, thereby preventing access of moisture into the U-tube when not in use or whilst being weighed.

A weighed quantity of the substance is introduced into the bulb, and is heated strongly, a current of pure dry air being at the same time aspirated through the tube. All moisture is drawn over into the exit U-tubes, the increase in weight of which represents the amount of water in that quantity of the substance operated upon.

#### Estimation of Carbon Dioxide.

**By direct Weighing.**—The carbon dioxide is liberated by treatment with a dilute acid, with the assistance of more or less heat,



FIG. 15.—SCHROTTER APPARATUS.

and is passed through a series of tared U-tubes, containing caustic potash or soda-lime, by suction from an aspirator. After complete

evolution the vessels are washed by means of a slow current of pure dry air or oxygen, in order that all the carbon dioxide may be drawn into the absorbing medium.

**By Difference.**—This method gives excellent results with careful manipulation, a very convenient form of the apparatus being that known as the Schroetter. This consists, as will be seen, in an arrangement made of thin glass, combining in one the acid reservoir, evolution chamber, and absorption cylinder. The reservoir is filled preferably with dilute hydrochloric acid (1 : 1), and the absorption chamber half filled with strong sulphuric acid to arrest any escaping aqueous vapour that may be carried along with the carbon dioxide. The vessel is accurately weighed, and 1 gramme of the substance being introduced into the lower chamber, the stopper having been slightly greased with a little vaseline, is set in firmly, and the dilute hydrochloric acid is run in slowly in small quantities at a time. When effervescence ceases, the apparatus is gently heated by a small burner over wire gauze in order to complete the reaction. A current of pure dry air is next aspirated through the apparatus by attaching a piece of rubber tubing to the exit stopper tube, in order to wash the chamber free of carbon dioxide ; the apparatus is then cooled in a desiccator and weighed. The loss in weight represents carbon dioxide.

#### Estimation of Iron.

The iron is precipitated as hydrate and weighed as oxide. For this purpose the solution must first be oxidized with nitric acid.

Dissolve 1 gramme pure recrystallized ferrous ammonium sulphate ( $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ ) in 100 c.c. of water, add a few drops of strong nitric acid, and boil the solution for several minutes. Care must be taken that the iron is completely oxidized. This may be seen by testing a drop of the assay solution with a drop of a freshly prepared solution of potassium ferrocyanide, when no blue colour appears ; otherwise more acid must be added, and the solution again boiled till this stage is reached. Now add excess of ammonium hydrate (specific gravity 0.880) to the hot solution, and again boil. Allow to settle, decant off the colourless supernatant liquor through a filter, wash the precipitate three times by decantation with hot water, afterwards transferring it to the filter, and continue washing

it with hot water till the wash water gives no precipitate with barium chloride. Dry the precipitate in the water-oven, detach it carefully from the filter, ignite the filter, and add the ash to the precipitate in a porcelain crucible. Heat both together at a red heat until constant in weight. From the weight of ferric oxide ( $\text{Fe}_2\text{O}_3$ ) the amount of iron in the amount taken may be got by multiplying by the factor 0·700.

#### Estimation of Aluminium.

Aluminium may be estimated as hydroxide in a similar manner, without the necessity, as above, of oxidizing. The precipitate is ignited and weighed as alumina ( $\text{Al}_2\text{O}_3$ ), and may be reported as such or as aluminium by multiplying by the factor 0·53015.

#### Estimation of Calcium.

If the calcium exists in an insoluble form, as in gypsum, the salt must first be fused with fusion mixture in order to convert it into the state of carbonate.

Dissolve 1 gramme pure calcium carbonate—Iceland spar suits admirably—in a little dilute hydrochloric acid, and dilute the resulting solution to about 100 c.c. with water. Heat, add ammonium hydrate in slight excess, and then a solution of ammonium oxalate, also in slight excess. Stir well, allow to settle, decant off the clear liquid through a quick filter, wash the precipitate three times by decantation, transfer it to the filter, and again wash it till the washings give no precipitate with silver nitrate. Dry in the water-oven, detach the precipitate completely from the paper, and place it in a porcelain crucible. Ignite the filter, and add its ash to the precipitate. Ignite at a gentle temperature, and finally for about twenty minutes at a bright red heat until the weight is constant. Weigh as  $\text{CaO}$ .

#### Estimation of Magnesium.

The magnesium is precipitated as magnesium ammonium phosphate ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ), ignited and weighed as magnesium pyro-phosphate ( $\text{Mg}_2\text{P}_2\text{O}_7$ ).

Dissolve 1 gramme pure magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in

about 50 c.c. cold water (note : this precipitation must be carried out with cold solutions), add a little ammonium chloride, and then excess of a solution of sodium phosphate. Stir well for at least fifteen minutes, and allow to stand for twelve hours. A rapid precipitation may be accomplished by shaking the assay solution in a stoppered cylinder for at least ten minutes, and as the precipitation is by no means very rapid, a prolonged period of settling is thus obviated. When a number of analyses are required to be carried out, a mechanical agitator will be found most useful.

The clear liquid is next decanted through a filter, and the precipitate washed with dilute ammonia (1 : 3) till free from sulphates, as shown by testing a few drops with barium chloride.

The precipitate is dried in the water-oven, ignited, until perfectly white and constant in weight, over the blow-pipe or in a muffle furnace at white heat. The magnesium is found by multiplying the weight of magnesium pyrophosphate thus obtained by the factor 0·21848.

### Estimation of Potassium.

The potassium is precipitated and weighed as potassium platinic chloride.

Dissolve 0·25 gramme pure potassium chloride in a little water acidified with a few drops of hydrochloric acid in a porcelain evaporating basin ; add at least three times its weight of platinic chloride dissolved in water, and evaporate down to almost dryness.

Excess of platinic chloride will be demonstrated by a yellow colour of the supernatant liquor.

Drench the crystals with a little alcohol, stirring them up gently with a glass rod. Allow to settle, filter through a tared filter, wash with alcohol till free from platinic chloride. This will be observed by the washings becoming colourless. Dry the filter, and precipitate in the water-oven until constant in weight. The weight of the potassium platinic chloride precipitate multiplied by 0·16091 equals K.

### Estimation of Phosphate (expressed as $\text{PO}_4$ ).

The phosphate is precipitated as magnesium ammonium phosphate ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ), ignited and weighed as magnesium pyro-phosphate.

Dissolve 1 gramme pure sodic phosphate ( $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ ) in 100 c.c. water, render slightly alkaline with ammonia, and add an excess of magnesia mixture. Stir well, and proceed as in the estimation of magnesium as phosphate.

The weight of ignited precipitate thus obtained multiplied by 0.63784 equals phosphoric anhydride ( $\text{PO}_4$ ).

Magnesia mixture is prepared by dissolving 180 grammes  $\text{MgSO}_4$  in 1,250 c.c. water, and dissolving in this solution 200 grammes ammonium chloride, finally adding 750 c.c. ammonia (specific gravity 880), and allowing it to settle for a few days.

Working upon 2 grammes of a phosphate, 50 c.c. of this solution will precipitate 100 per cent. tricalcic phosphate.

#### Estimation of Sulphuric Acid in a Soluble Sulphate.

The sulphate is precipitated with barium chloride, and weighed as barium sulphate.

For this purpose dissolve 1 gramme pure copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in 100 c.c. water, acidify with a few drops of hydrochloric acid, boil, add a little ammonium chloride and excess of barium chloride (hot solution), and boil for some time. Allow to settle, wash by decantation with hot water, then transfer the precipitate to the filter, continuing the washing till free from chlorine (indicated by testing the washings with silver nitrate). Dry, detach the precipitate from the filter, incinerate the paper, add it to the precipitate in a porcelain crucible, and ignite it at a red heat until constant in weight.

Any barium sulphate on the paper which may be reduced in the incineration to sulphide may be oxidized to sulphate by adding 1 drop of hydrochloric acid along with 2 drops of sulphuric acid, gently expelling excess of acid at a low temperature, and igniting at a red heat. Finally, weigh as barium sulphate.

## CHAPTER IV

### VOLUMETRIC ESTIMATIONS

#### Note on Volumetric Analysis.

VOLUMETRIC analysis consists chiefly in applying accurately measured volumes of reagents to known volumes of the solutions of the substance to be tested, both solutions being of known strength, and thereby bringing about certain chemical changes, the results of which are visible to the naked eye, either alone or with the assistance of secondary reagents termed "indicators."

Gravimetric analysis generally requires a considerable length of time, together with more or less apparatus, and very often becomes very complicated and tedious. These difficulties are mostly, if not in all cases, overcome by the use of volumetric methods which combine simplicity with accuracy, embracing the use of the solution of the substance to be tested, the "standard" solution of the reagent, the indicator (if required), and the measuring vessel or "burette."

All measuring vessels must be accurately graduated; the standard solutions must be accurate as to strength and purity, and must agree with each other.

There are three chief classifications of volumetric methods:

1. The substance is estimated by saturating its solution with a standard solution of exactly opposite chemical properties—*e.g.*, acid and alkali.
2. The substance is estimated by precipitation, and noting the volume of standard solution of the precipitant that may be required, such as silver nitrate and sodium chloride solutions.

3. The substance is oxidized—*e.g.*, by  $\text{KMnO}_4$ ,  $\text{K}_2\text{CrO}_4$ —or reduced ( $\text{SnCl}_2$ ,  $\text{Na}_2\text{S}_2\text{O}_3$ ), the end-point being made clearer and

sharper by the use of an indicator, and the volume of standard solution of the agent used noted, such as in the estimation of iron by potassium bichromate.

### Indicators.

Most indicators are less sensitive in hot liquids than they are in the cold.

**Litmus.**—This solution is prepared by repeatedly treating solid (cube) litmus with boiling water. The combined extractions are then stood over for two or three days to settle ; the clear liquid is then decanted off and preserved in bottles with plenty of access of air. The addition of 2 or 3 drops of chloroform assists its preservation. Litmus cannot be used in the presence of carbonic acid ; its end-point is sharp, and is indeed one of the most sensitive of indicators.

Litmus-paper is prepared by soaking strips of filter-paper in litmus solution (red and blue) and allowing them to dry slowly.

**Cochineal.**—Unlike litmus, this indicator is not affected by carbonic acid, and it may be used in gaslight. Its solution is prepared by dissolving 1 grainne of powdered cochineal in 20 c.c. alcohol and diluting to 100 c.c. with water. The clear solution is reddish-yellow, changing to violet-red with alkalies.

It cannot be used in presence of salts of iron, alumina, or acetic acid.

**Turmeric.**—This is not a very satisfactory indicator. A slightly alkaline alcoholic tincture of turmeric may be used for preparing turmeric paper in the titration of organic acids by alkalies.

**Methyl Orange** (para-dimethylaniline-azo-benzene-sulphonic acid) is an orange powder soluble in water, a suitable strength being 1 in 1,000. It is very sensitive, especially to heat, and must not be used in excess, else the end-point is slow and uncertain. It may be used in presence of carbonic, hydrocyanic, boric, arsenious, stearic, and carbolic acids, and sulphuretted hydrogen, but not with organic acids, as acetic, citric, oxalic, etc. It is, generally speaking, the most convenient indicator.

**Phenolphthalein** (dioxydiphenyl-phthalide).—One gramme may be dissolved in 100 c.c. alcohol of 50 per cent. It cannot be used

for ammonia or its compounds, but is specially useful in titrating the organic acids.

**Erythrosin.**—This indicator is extra sensitive, and may be used for the estimation of minute quantities of alkalies or acids. It is turned to a rose colour by alkalies.

### Standard Solutions.

**Normal Soda Carbonate** (53 grammes  $\text{Na}_2\text{CO}_3$  per 1,000 c.c.).—Ignite about 60 grammes pure anhydrous soda carbonate in a platinum dish at a dull red heat for about twenty minutes. Cool in a desiccator, weigh out exactly 53 grammes, place the salt in a litre flask, dissolve in water, and make up to the mark at  $16^\circ \text{ C}$ . This solution will keep indefinitely if made with distilled water.

**Normal Sulphuric Acid** (49 grammes  $\text{H}_2\text{SO}_4$  per 100 c.c.).—Mix 35 c.c. pure sulphuric acid (specific gravity, 1.840) with about four times its volume of water by pouring the acid gradually into it with gentle shaking. Allow to cool, and make up to 1,000 c.c. at  $16^\circ \text{ C}$ .

The solution is now ready for standardization against the normal alkali solution above.

Clean out a burette, first with soap and water, to free it from all grease, and then with hot water, and finally cold water. Always rinse out the burette with a little of the solution before proceeding to fill it up to the zero mark, allow it to drain, and then fill up to the mark. An Erdmann float is very useful, especially when the illumination is defective. Run out 10 c.c. into a beaker, dilute to 50 c.c., and estimate the sulphuric acid gravimetrically by barium chloride. Then pipette 20 c.c. of the  $\text{Na}_2\text{CO}_3$  solution into a beaker, and run in sulphuric acid from the burette until the neutral point is reached, using methyl orange as an indicator. The acid must then be diluted or strengthened until 20 c.c. of the acid exactly equal 20 c.c. of the alkali solution.

### Weights and Factors required in Volumetric Analysis.

A normal solution is one that contains in 1 litre that proportion of its molecular weight in grammes of the substance which corresponds to 1 gramme of available hydrogen or its equivalent.

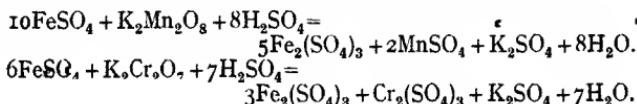
Thus,  $\frac{1}{2}$  (normal) hydrochloric acid contains 36·5 grammes HCl, and  $\frac{1}{2}$  sulphuric acid  $\left(\frac{98}{2}\right)$  = 49 grammes H<sub>2</sub>SO<sub>4</sub>.

Normal acid solutions are equivalent in strength to normal alkali solutions.

Normal sulphuric acid ..	1 c.c. = 0·049	gramme H <sub>2</sub> SO <sub>4</sub> .
Normal hydrochloric acid ..	1 c.c. = 0·0365	gramme HCl.
Normal nitric acid ..	1 c.c. = 0·063	gramme HNO <sub>3</sub> .
Normal oxalic acid ..	1 c.c. = 0·063	gramme H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> .
Normal acid ..	1 c.c. = 0·017	gramme NH <sub>3</sub> .
	= 0·035	gramme NH <sub>4</sub> OH.
	= 0·028	gramme CaO.
	= 0·056	gramme KOH.
	= 0·04	gramme NaOH.
	= 0·053	gramme Na <sub>2</sub> CO <sub>3</sub> .
	= 0·084	gramme NaHCO <sub>3</sub> .
Normal sodium hydrate ..	1 c.c. = 0·040	gramme NaOH.
Normal potassium hydrate ..	1 c.c. = 0·056	gramme KOH.
Normal soda carbonate ..	1 c.c. = 0·053	gramme Na <sub>2</sub> CO <sub>3</sub> .
Normal alkali ..	1 c.c. = 0·035	gramme B <sub>2</sub> O <sub>3</sub> .
	1 c.c. = 0·062	gramme H <sub>3</sub> BO <sub>3</sub> .
	1 c.c. = 0·088	gramme bityric acid.
Decinormal silver nitrate ..	1 c.c. = 0·0108	gramme silver.
	1 c.c. = 0·00355	gramme chlorine.
Decinormal iodine ..	1 c.c. = 0·00495	gramme As <sub>2</sub> O <sub>3</sub> .
Decinormal bichromate ..	1 c.c. = 0·02456	gramme FeSO <sub>4</sub> .
	1 c.c. = 0·0216	gramme Fe.
Decinormal thiosulphate ..	1 c.c. = 0·0248	gramme Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .5H <sub>2</sub> O.
	= 0·0127	gramme I.
	= 0·00355	gramme Cl.
	= 0·0080	gramme Br.

### Estimation of Iron.

This operation consists in oxidizing the iron, previously brought into the ferrous condition by treatment, if necessary, with such reducing agents as sulphurous acid or, better still, stannous chloride, into the ferric state by treatment with potassium permanganate or bichromate at ordinary temperatures in presence of free sulphuric or hydrochloric acids, but not acetic acid, according to the equation



**Solution of Potassium Permanganate.**—Dissolve 2·82 grammes of the pure salt in water, and dilute the solution to 1,000 c.c. Then 1 c.c. should be equal to 0·5 grammes Fe. To ascertain the exact strength of the solution it must be standardized (from time to time, as it is unstable) against a solution prepared by dissolving 0·2 grammes of pure iron wire (piano wire suits) in 10 c.c.  $H_2SO_4$ , and diluting to 1,000 c.c. and titrating. The solution may also be standardized against a correct solution of oxalic acid. The permanganate titration is more advantageous to work with in that it is easier, and the end-point is sharper. The solution, however, is unstable, and must not be used in presence of hydrochloric acid or free chlorine, excess of nitric acid or organic matter.

**Solution of Bichromate.**—Dissolve 4·913 grammes of the pure dry salt in water, and dilute to 1,000 c.c.

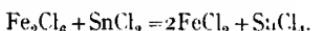
**Estimation of the Iron in Ferrous Solutions.**—Weigh out 1 grammme pure recrystallized ferrous ammonium sulphate ( $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ ), dissolve it in water, acidify with a few drops of sulphuric acid, and make up to 250 c.c. Mix thoroughly, and pipette off 25 c.c. into a clean beaker. Dissolve 0·1 grammme potassium ferricyanide ( $K_3Fe(CN)_6$ ) in 100 c.c. water, and spot out drops of equal size of this solution over the surface of a clean white tile at equal distances, and in line with one another. Spot plates—that is to say, porcelain tiles having suitable cup-shaped depressions—may be had for this purpose. A trace of a ferrous salt will turn these spots blue, ferric salts exhibiting no visual change.

Run in the bichromate solution from a burette, about 1 c.c. at a time, into the warm ferrous solution (at about 60° C.), stir well, and after each addition lay out a drop in contact with 1 or 2 drops on the plate. More bichromate is then added until a precipitate appears. This is at first greenish in colour. The solution must now be added very carefully, a few drops at a time, until the assay gives with the test a colour which is faintly greener than two drops of the assay solution placed alongside. The operation is then repeated with a fresh quantity of the ferrous solution until concordant results are obtained, in which case the bichromate may be run in in a steady stream to within a drop or two of the endpoint.

**Calculation.**—The total amount of iron in the 250 c.c. is obtained

by multiplying the number of c.c. of bichromate used by the factor 0·0056, and then by 10, as 25 c.c. were taken at each operation.

**Estimation of Iron in Ferric Solutions.**—The iron is reduced to the ferrous state by means of a suitable reducing agent, such as stannous chloride, according to the equation



Dissolve 20 grammes stannous chloride in 20 c.c. dilute hydrochloric acid, and dilute the solution to 1 litre. Fill a burette with this solution, cautiously run this into the hot ferric solution (which has been previously obtained by solution in hydrochloric acid) until the colour is discharged. This will necessitate excess of stannous chloride. A large excess must be avoided, which must then be oxidized by adding a small quantity (1 to 5 c.c.) mercuric chloride (2·5 per cent.  $\text{HgCl}_2$ ). Boil; if the solution clears or only a very slight precipitate of inercurons chloride remains, it may be cooled and titrated as described before with the bichromate solution.

#### Estimation of Chlorine.

Dissolve 16·960 grammes pure recrystallized silver nitrate in distilled water, and make the solution up to 1 litre. Store the solution in a dark blue glass-stoppered bottle in a dark place. Prepare a 2 per cent. solution of potassium chromate free from chlorine.

*Process.*—Weigh out 1 grainme pure common salt, dissolve in water, and make the solution up to 250 c.c. Pipette out 25 c.c. of this solution into a porcelain evaporating basin for titration, and a drop or so of the potassium chromate. Run in from a burette the silver nitrate solution; at each addition of silver nitrate a red precipitate of silver chromate will form, which will dissolve in the excess of the chloride solution with the aid of a little stirring. A white fugitive precipitate of silver chloride may also be noticed. On the continued addition of the silver nitrate the precipitate will remain undissolved, settling to the bottom, and leaving a yellow supernatant liquid. At this stage continue adding more silver nitrate very cautiously, drop by drop, until the yellow liquid just changes to a reddish tinge. Repeat the experiment, running in the

silver nitrate in a steady stream, to within a few drops of the end-point.

Each c.c. silver nitrate corresponds to 0.003545 grammes chlorine, or 0.005845 grammes sodium chloride.

#### **Estimation of Phosphoric Acid (Uranium Process).**

This process is based upon the fact that, when a solution of uranium nitrate or acetate is added to a solution of a phosphate in the presence of an alkaline acetate and free acetic acid, the whole of the phosphoric acid is thrown down as uranium phosphate ( $\text{Ur}_2\text{O}_3 \cdot \text{P}_2\text{O}_5$ ) in the form of a yellow precipitate. A solution containing free mineral acid must be neutralized, and excess of the alkaline acetate added, together with excess of acetic acid. The excess of uranic solution is ascertained by testing a drop of the assay solution with 1 drop of freshly dissolved potassium ferrocyanide.

The solutions required are—

1. S.S. Uranium nitrate or acetate : 1 c.c. equals 0.005 grammes  $\text{P}_2\text{O}_5$ . Weigh out about 35 grammes of the salt, acidify with 25 c.c. glacial acetic acid, dissolve, and dilute to 1 litre with distilled water.

2. S.S. Tribasic phosphate : 50 c.c. equals 1 grammes  $\text{P}_2\text{O}_5$ . Dissolve 5.886 grammes sodium ammonium phosphate (previously well dried by pressing between filter-paper) in water, and make the solution up to 100 c.c.

3. Solution of sodium acetate, prepared by dissolving 100 grammes sodium acetate in water, adding 50 c.c. glacial acetic acid, and diluting to 1 000 c.c.

4. Solution of potassium ferrocyanide (freshly prepared).

*Process.*—Pipette 50 c.c. of the sodic phosphate solution into a beaker, add 5 c.c. sodic acetate solution, and heat to 90° C. Run in the uranic solution from a burette in slightest excess—that is to say, until 1 drop of the assay solution produces with 1 drop of the solution of ferrocyanide the faintest reddish-brown coloration. Once more heat the assay solution to 90° C., and again “spot out.” If the coloration persists, the assay is completed; if the coloration fails, more uranium must be added to produce this tint.

The uranium solution is then diluted until 20 c.c. equal 50 c.c. of the phosphate solution. The solution is finally checked by running in rapidly the uranium solution to within a few drops, and then finishing off very accurately.

### Estimation of Phosphoric Acid in Bones, Soluble Phosphates, etc.

(*In Solutions Free from Iron and Alumina.*)

The uranium solution for this purpose is standardized against a standard solution of tricalcic phosphate. Dissolve 50 grammes pure tricalcic phosphate in a small quantity of dilute hydrochloric acid, then add a slight excess of ammonia, sufficient to cause precipitation, which redissolve in a slight excess of acetic acid, and dilute to 1,000 c.c. with distilled water. It is advisable to run the uranium solution into a known quantity of the phosphate solution. In the event of the tricalcic phosphate salt not being pure, it is well to estimate the phosphoric acid in it by dissolving in a little nitric acid, precipitating with slight excess of ammonium molybdate, redissolving the washed phospho-molybdate precipitate in ammonia, and finally estimating the phosphoric acid as magnesium pyrophosphate with magnesium mixture. If the salt is found pure, a quantity equal to 5 grammes is dissolved as above, and diluted to 1 litre.

**S.S. Uranium Acetate.**—This should be of such a strength that 25 c.c. equal 50 c.c.

**S.S. Calcic Phosphate.**—Then 1 c.c. S.S. uranium equals 1 per cent. soluble tricalcic phosphate, using 1 gramme of the sample.

**Process.**—For superphosphates, etc., containing only traces of iron and alumina, take 10 grammes of the sample in a mortar, and crush gently with a little cold water to a creamy consistency. Transfer this cream to a flask graduated at 503·5 c.c.; the excess (3·5 c.c.) allows for the presence of any insoluble matter in a commercial superphosphate, running from 25 to 30 per cent. Dilute to the mark with cold water, and shake occasionally for half an hour. Filter through a dry paper into a dry beaker, and measure out accurately 50 c.c. (equal to 1 gramme sample) of the clear filtrate into another beaker for titration. Add a 10 per cent. solution of

ammonia in just sufficient quantity to precipitate the monocalcic phosphate as tricalcic phosphate, redissolve this precipitate in a slight excess of acetic acid. The presence of iron and alumina will be seen at this stage by the solution remaining opalescent ; with only a slight opalescence the process may be proceeded with, but with more than a trace the operation must be stopped, and the sample treated specially, as hereafter described, in order to eliminate the iron and alumina. The uranium solution is now run into the cold solution with stirring, until a faint chocolate colour is observed with the ferrocyanide upon the spot plate. The assay solution is then heated to 90° C., and the titration finished at that temperature. A second titration is carried out by running in nearly all the uranic solution to within a few drops of the end-point in a steady stream, this titration being further checked by a third assay.

- In cases where the iron and alumina and other substances occur to such an extent that the accuracy of the process as above described is interfered with, the phosphoric acid must be precipitated with ammonium molybdate solution in nitric acid solution, as described in the gravimetric section, washed, dissolved in excess of ammonia, and reprecipitated by magnesia mixture, redissolved, and finally titrated with the uranium solution. Or the solution may be treated direct by Joulie's citro-magnesia method as follows :—

**Joulie's Citro-Magnesia Process.**—Treat from 1 to 10 grammes of the sample with hydrochloric acid, or if necessary with hydrochloric and nitric acids, in a measuring flask till all but silica remains, cool, make up to the mark, and filter through a dry filter. Of the clear filtrate pipette out a portion equivalent to about 0·05 gramme of phosphoric acid, and to it add sufficient of the citro-magnesic solution to form a precipitate, which will only appear after a few moments. For this purpose add 10 c.c. of the citro-magnesic solution. A direct precipitate will show the presence of phosphates of iron and alumina. More solution must be added, not to the assay solution, but to a fresh similar portion of the phosphate filtrate, as the precipitated iron and aluminium phosphates are rather difficult to redissolve. A fresh portion—say 20 c.c.—of the citro-magnesic solution may be added, in order to effect slow precipitation.

**Citro-Magnesic Solution.**—Slowly dissolve by gradual additions 27 grammes magnesia carbonate in 270 grammes citric acid dissolved in 350 c.c. hot water. After complete solution, cool, and add 40 c.c. concentrated ammonia; dilute to 1 litre.

**S.S. Uranium Nitrate.**—Dissolve 40 grammes of this salt in 800 c.c. water, render slightly turbid with ammonia, redissolve in acetic acid, and dilute to 1 litre. Standardize this solution against a standard solution containing 8·10 grammes pure acid ammonium phosphate per 1,000 c.c. Each c.c. of this solution will equal 0·005  $P_2O_5$ .

*Process.*—The weighed quantity of the sample having been dissolved and filtered free from any silica, the phosphates are precipitated in the form of ammonium magnesium phosphate, allowed to stand for about twelve hours, filtered, washed with dilute ammonia till free from magnesia, redissolved in dilute nitric acid, brought slightly back with ammonia, and again just dissolved in dilute nitric acid (this to avoid any excess of acid) heated to 90° C. Five c.c. acetate of soda solution added, and titrated with S.S. uranic nitrate.

#### Pemberton's Method.

**Solution of Ammonium Molybdate.**—Dissolve 90 grammes in 700 c.c. water, allow to stand. Any precipitate of molybdic acid may be redissolved after decantation in a little ammonia. Make the clear solution up to 1 litre. Each c.c. will precipitate 3 milligrammes  $P_2O_5$ .

**S.S. Sulphuric Acid.**—Dilute 323·7 c.c. normal acid to 1 litre.

**S.S. Potassium Hydrate.**—Prepare a solution of like strength, and check it against the acid, using as an indicator phenolphthalein.

**S.S. Ammonium Nitrate.**—A saturated aqueous solution.

*Process.*—Dissolve 1 grammie of the phosphate in a little nitric acid, filter, cool, dilute the filtrate up to 250 c.c. with water. • Pipette 25 c.c. of the filtrate into a beaker, neutralize with ammonia, add 5 c.c. concentrated nitric acid, 10 c.c. ammonia nitrate, and dilute to 65 c.c. Boil, add 5 c.c. molybdate solution with constant stirring, allow to settle, repeating the operation until no further precipitate is formed with more of the molybdate solution. It will be found that from 10 to 15 c.c. will be required as a rule. A

large excess of the molybdate, however, ought to be avoided. Filter through a 7-centimetre filter, washing the precipitate on to the filter by decantation with water; wash further with water, and transfer it back into the same beaker, and run in from a burette

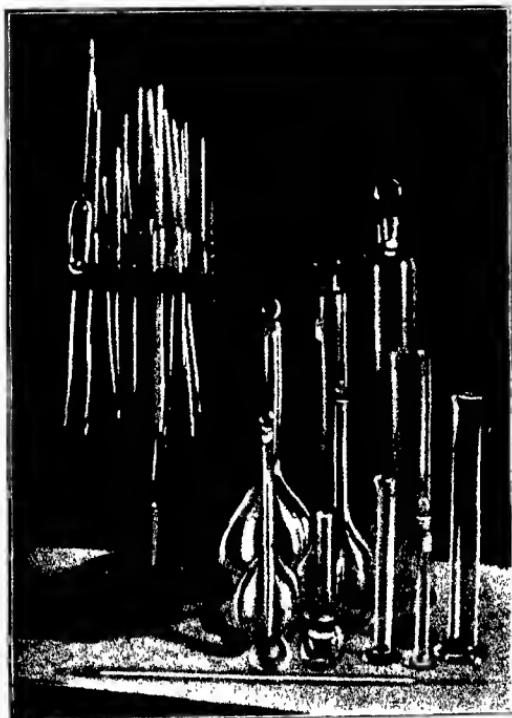


FIG. 10.—MEASURING APPARATUS : FLASKS, CYLINDERS,  
AND PIPETTES.

sufficient S.S. KOH to dissolve the precipitate. Add phenolphthalein, and neutralize with S.S. sulphuric acid.

Subtract the amount of acid used in neutralization from that of the alkali used for effecting solution, the remaining amount of alkali being equal to the direct percentage of phosphoric anhydride present, each c.c. alkali being equal to 1 per cent.  $P_2O_5$ , and

adoperating upon 1 gramme of the sample. Thus, supposing 31 c.c. of alkali were used to neutralize the free phosphoric acid, the actual percentage of  $P_2O_5$  in the sample would be 31 per cent.  $P_2O_5$ .

### Soluble Phosphates in Superphosphates, etc.

The soluble phosphoric acid is obtained in an aqueous extraction of the sample, an aliquot part of which is taken, and is proceeded with as already described for total phosphate.

This process is rapid, occupying less than one hour, and gives excellent results, with a tendency, if anything, to slightly high assays.

## CHAPTER V

### ANALYSIS OF SOILS

**Sampling.**—The sample should in all cases be typical of the nature of the soil to which it belongs, the method of sampling depending upon the subsequent form of analysis, whether geological, mineralogical, or agricultural.

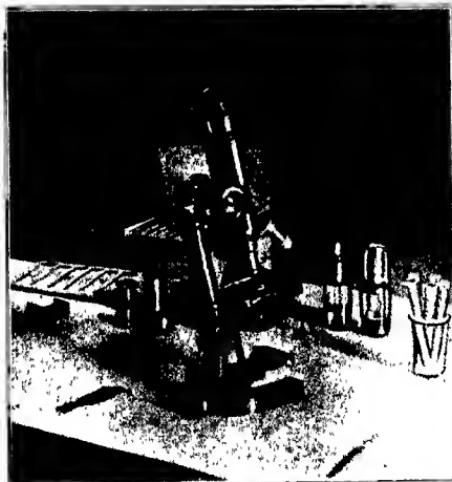


FIG. 17.—MICROSCOPE AND ACCESSORIES.

To great care, therefore, cannot be taken in order to obtain uniform samples, as the analyses of soils are long and expensive. A suitable spot having been selected, a square hole of about 18 inches deep is dug out with a spade. A sample of 6 to 9 inches deep and 4 inches thick, is cut out from one of the sides and laid aside, together with

any particles that may have fallen down into the bottom of the hole in the cutting. The subsoil is sampled in a similar manner. It is advisable to select several places in one field, mixing the subsamples thus obtained, and finally return to the laboratory a sample of about 10 pounds weight.

It is seldom found necessary to take a sample from a depth of more than 6 feet or so. In taking such a sample, a special sampling apparatus, such as a large special auger, is very useful.

Samples should preferably be taken after harvest and before manuring, the nature of débris, stones, etc., and geological formation being carefully noted.

The sample is broken down on a sampling bench with the fingers, and carefully freed from all stones, wood, and insects, the loss in weight being found. It is then passed through a 1-millimetre sieve.

**Physical Tests—Colour.**—The colour of a soil depends largely upon the amount of organic matter and iron compounds present. Soils rich in organic matter are more or less dark when in a moist condition. Iron colours soil a red colour. Dark or red soils are the most fertile. The colour is best observed when heating the sample in an oven up to  $110^{\circ}$  C.

**Specific Gravity.**—The true specific gravity of soils is about 2.5. Ten grammes of the dry soil ( $100^{\circ}$  C.) are boiled till air-free with a small quantity of water, and introduced into a specific-gravity bottle, all particles being carefully transferred with the aid of fresh distilled water. The bottle is filled with water, cooled, and weighed as usual. Alcohol may be used in place of water in cases where there is a water soluble content in the sample.

Weight of bottle .. .. .. .. ..	<i>y</i>
Weight of bottle with distilled water at $15.5^{\circ}$ C. .. .. .. .. ..	<i>x</i>
Weight of water in bottle .. .. .. .. ..	<i>x - y</i>
Weight of sample taken .. .. .. .. ..	<i>z</i>
Weight of bottle, sample, and water .. .. .. .. ..	<i>a</i>
Weight of sample and water .. .. .. .. ..	<i>a - y</i>
Weight of water .. .. .. .. ..	$(a - y) - z$
Weight of water displaced by 10 grammes ample .. .. .. .. ..	$(a - \frac{y}{2}) - \{(a - y) - z\} = \theta$
Therefore specific gravity equals $10.00 \div \theta$	

**Soil Temperature.**—The method of Frear is to arrange a series of thermometers in a trench at various depths, varying from 1 to 24

inches, or in rocks from 5 to 7 feet. Readings are taken thrice daily.

**Mechanical Analysis—Sifting.**—The soil is passed through a series of sieves made by punching circular holes in a sheet of zinc. The sieves are placed in the form of a battery, the first sieve having holes 2 millimetres in diameter; the second sieve having holes 1 millimetre in diameter; the third sieve having holes 0·5 millimetre in diameter. In some cases the soil is apt to adhere to the sieve, so that it requires to be assisted with a little water.

**Chemical Analysis of Soils.**—The sample is passed through a 1-millimetre sieve, and air-dried till constant in weight. Ordinary arable soil requires about four days, rich dark soil about six days, and peaty or mossy soils about twelve days. The sample is then examined as to its reaction with litmus, its hygroscopicity, combined water, organic matter, carbon dioxide (existing as carbonates),  
•and the portions soluble and insoluble in acids.

**Reaction.**—This is made as—

1. Water eliminated at ordinary temperatures—*i.e.*, air-dried.
2. Water eliminated at 110° C.
3. Water eliminated at red heat.

Ten grammes are accurately weighed out into a shallow dish, and dried in air for several days until constant in weight.

Five grammes of the air-dried soil are placed in a shallow platinum dish and dried in an air-bath at 110° C. for eight hours, cooled in a desiccator, and weighed, the operation being repeated until the weight is constant, the subsequent heatings extending to one hour each time. Care must be taken to weigh rapidly. The platinum dish containing the 5 grammes of soil used in determining the hygroscopic moisture is heated to a dull redness until all organic matter is expelled, care being taken not to raise the temperature sufficient to volatilize any inorganic salts (alkaline chlorides), cooled, and then drenched with a few drops of a saturated solution of ammonium carbonate, dried, and again heated to 150° C. to expel excess of ammonia.

The loss thus sustained is due to organic matter, combined water, and ammoniacal salts.

An alternative method is to heat the residue from (2) in a com-

bustion tube, and aspirate the moisture evolved through a series of tared U-tubes containing calcic chloride.

**Direct Estimation of Carbon.**—Ten grammes of the soil are ground in an agate mortar to a very fine powder, placed in a flat-bottomed dish, and covered with a thin layer of sulphurous acid, with constant stirring. The carbonates are thus eliminated; the dish is then dried, and its contents, when finely ground, are placed in a platinum boat, and ignited in a current of oxygen in a combustion tube about 20 inches long, the front portion of which is filled for at least 8 inches with cupric oxide. The exit end of the tube is drawn out, and is connected with a series of absorption tubes. The first tube contains calcic chloride, the second glass moistened with concentrated sulphuric acid, and the last two soda-lime for the purpose of absorbing the carbon dioxide.

The combustion is carried out in the usual way, by first heating the cupric oxide to a red heat, and passing the oxygen through for some time.

According to Wolff, the humus in the soil contains 58 per cent. carbon. If the percentage of carbon thus found be multiplied by 1.724, or the carbon dioxide by 0.471, the figure obtained represents the amount of humus in the soil.

**Estimation of Organic Hydrogen.**—The total hydrogen is estimated by ignition in a current of oxygen, and weighing the water thus obtained in absorption tubes. This consists of pre-existing water and organic hydrogen, the amount of the latter being found by subtracting the total moisture (free, hygroscopic, and combined) from the total water thus produced.

**Estimation of Organic Oxygen.**—This is obtained indirectly, by calculation, from the oxygen in the proteid and ligneous matters, according to the formula of Berthelot and André :

Let  $\phi$  equal the weight of proteids found,

Then O equals  $\frac{\phi \times 35.5}{100}$ .

Let  $\phi'$  equal the weight of ligneous matter,

Then O' equals  $\frac{\phi' \times 49.4}{100}$ .

Therefore the total oxygen (approximately) equals O + O'

**Estimation of Humus.**—Coarse sand is well washed with acids and ignited. It is then intimately mixed with 10 grammes of the finely divided soil, and placed in a small funnel, the neck of which is filled with small, broken glass.

The mixture is digested with a small quantity of equal parts of ammonia and distilled water for three hours. The alkali dissolves the dark matter. The solution is tapped off, and the residue further treated with distilled water. The solution is filtered and evaporated to dryness in a platinum dish, weighed, and expressed as humus. The residue is ignited, and the phosphoric acid determined in the ash.

**Estimation of Free Humic Acid.**—This is of importance in ascertaining the amount of lime required for acid soils.

Twenty grammes of the finely divided sample are digested with 50 c.c. strong ammonia for two or three days in a warm place. The bulk is made up to 1 litre, thoroughly agitated, and allowed to settle for twenty-four hours; 500 c.c. of the supernatant liquor is acidified with hydrochloric acid to precipitate the humus, which is then collected on a tared filter, dried, and weighed. It is next ignited and the ash deducted from the first weight, thus giving the free humic acids freed from inorganic salts.

**Estimation of Carbonates.**—Five to 50 grammes of the soil are rubbed up in a porcelain mortar or basin with freshly distilled water into a thin pasty consistency. All air is expelled by thorough kneading and stirring. The mixture is made up to 300 c.c., and the carbon dioxide liberated by the addition of acid is absorbed in tared potash bulbs.

**Estimation of the Inorganic Content.**—Place 10 grammes of the air-dried soil in a round-bottomed flask of 150 to 200 c.c. capacity; add 100 c.c. pure hydrochloric acid (specific gravity, 1·11); cork the flask, and wire it down securely; digest it for thirty-six hours on a water-bath at 100° C.; filter, and wash well with hot water. The residue is that portion insoluble in hydrochloric acid. To the filtrate add a few drops nitric acid, and evaporate to dryness on the water-bath; treat the residue with hot water containing a few drops of hydrochloric acid, and again evaporate to dryness. Repeat the treatment with the hydrochloric acid water, and filter into a litre flask, washing the precipitate with hot water. Cool, and make up

to 1 litre. The precipitate is the silica. This is Solution A. Take 100 to 200 c.c. of Solution A, and add ammonia till alkaline. Expel excess of alkali by boiling, settle, decant through a filter, and wash by repeated decantations with hot water, passing the solution through the filter each time. Dissolve the precipitate in the beaker in the smallest possible quantity of warm hydrochloric acid, and add sufficient ammonia to precipitate the oxides of iron and alumina only. Again wash by decantation with hot water, and finally pour all the precipitate upon the filter, washing it till free from chlorine reaction.

The combined filtrates and washings are set aside as Solution B.

Dry the filter and contents at  $110^{\circ}\text{C}.$ , and ignite them apart in a platinum crucible at a red heat ; cool and weigh. This represents the oxides of iron and alumina and phosphoric acid.

*Ferric Oxide*.—Precipitate the iron as above in a fresh portion of 100 c.c. of Solution A by heating with ammonia. Filter, wash with hot water, dissolve in dilute sulphuric acid, reduce to the ferrous condition with zinc, and titrate with a standard solution of potassium permanganate. Calculate to ferric oxide.

*Alumina*.—Deduct the calculated weight of ferric oxide, filter ash and phosphoric acid as found below from the combined precipitate found above to obtain the weight of alumina.

*Phosphoric Acid*.—This is estimated in the iron solution by the ammonium molybdate method.

*Manganese*.—Concentrate the combined filtrates and washings forming Solution B to at least 200 c.c., render alkaline with ammonia, oxidize with a drop of bromine, and heat to boiling in a flask. The manganese thus precipitated is allowed to settle, is filtered whilst still warm, washed well with hot water, dried, ignited, and weighed as oxide.

*Lime*.—To the filtrate from the manganese add 20 c.c. strong ammonium chloride solution and 40 c.c. saturated ammonium oxalate. Heat to boiling, allow to settle, filter, and wash by decantation with hot water, finally transferring all the precipitate to the filter, and washing it till free from chlorides. Dry, ignite, and weigh as oxide.

*Magnesia*.—Concentrate the combined filtrates and washings from the lime to at least 200 c.c. ; make alkaline with ammonia (about 20 c.c.), and add slowly and with constant stirring 30 c.c.

of a saturated solution of sodium phosphate ; stir vigorously and at intervals, and then allow to stand over for twelve hours. Filter ; wash thoroughly with ammonia water (1 : 3) till free from phosphate ; dry, and ignite first at a gentle heat, and ultimately at a white heat ; cool, and weigh as magnesium pyrophosphate.

Magnesium pyrophosphate multiplied by 0.36203 equals MgO.

*Sulphuric Acid.*—Evaporate 200 c.c. of Solution A to nearly dryness on the water-bath. Take up in a small quantity of distilled water, and add 10 c.c. of barium chloride solution. Boil for at least five minutes, allow to settle, filter, and wash with hot water till free from chloride ; dry and ignite strongly : cool, and weigh as barium sulphate. Weight of precipitate multiplied by 0.34331 equals sulphuric anhydride in the soil.

*Potash and Soda.*—To a fresh portion of 200 c.c. of Solution A add barium chloride in slight excess, render alkaline with ammonia, and thus precipitate the sulphuric acid, ferric oxide, etc. Precipitate the lime and barium with ammonium carbonate ; evaporate the filtrate and washings to dryness, ignite at a dull red heat to expel ammoniacal salts, dissolve in a small quantity of distilled water. Filter, and wash precipitate. Combine filtrate and washings, add 10 c.c. barium hydroxide, and digest for at least one hour. Filter, wash precipitate, add ammonium carbonate to ensure complete precipitation of the barium, filter, and wash. Evaporate the filtrate and washings in a tared platinum dish, ignite gently to expel ammoniacal salts, cool, and weigh the combined chlorides of potassium and soda. Estimate the potassium, if necessary, by precipitation with platinic chloride.

**Examination of the Soil Insoluble in Hydrochloric Acid.**—The portion insoluble in hydrochloric acid is washed with hot water till free from acid reaction by decantation, the residue is transferred to a porcelain dish, dried, and weighed till constant in weight. The mass is thoroughly mixed, and a weighed portion is ignited in a platinum crucible. Two grammes are then taken in the crucible, and mixed with 12 grammes of ammonium fluoride, with the aid of a platinum rod, and gently heated over a Bunsen burner or in a small muffle for one hour. Moisten with 2 c.c. sulphuric acid, driving off excess of acid at a low temperature. Treat with hydrochloric acid water, and transfer to a 500-c.c. flask ; oxidize thoroughly

with nitric acid ; cool, and make up to 500 c.c. Proceed to estimate the constituents as in the portion soluble in hydrochloric acid.

**Method of Estimating Phosphoric Acid in Soils** (Carnot).—Ignite 10 grammes of the soil, to free it from organic matter ; cover with water, and then add nitric acid (100 c.c.), and digest at about 100° C. for two hours, with frequent stirring and additions of small quantities of acid. Filter, wash with hot water, and concentrate the combined filtrates and washings to 50 c.c. Add 5 c.c. concentrated nitric acid and 0·5 gramme solid chromic acid. Cover dish with a funnel, and continue boiling for thirty minutes. Add 5 grammes ammonium nitrate, and then 50 c.c. ammonium molybdate, and allow to stand for one hour at about 100° C. Wash twice by decantation with ammonium molybdate water (1 : 5). Dissolve in 30 c.c. dilute ammonia (1 : 1). The combined filtrate and washings, which should now measure 80 c.c., is neutralized with nitric acid, and the temperature kept well below 40° C. Stir well, and when the yellow precipitate ceases to dissolve add 3 c.c. nitric acid and 5 c.c. water, and a like volume of molybdate solution. Filter, wash with nitric acid water (1 : 100), and then with water, dry, and weigh till constant. Weight of precipitate multiplied by 0·0373 equals phosphoric acid.

**Estimation of Chlorine** (Wolff).—Macerate 300 grammes of the soil with 900 c.c. distilled water, acidified with a little nitric acid, with frequent shaking, for forty-eight hours. Filter off 500 c.c., and concentrate this to 200 c.c. Precipitate the chlorine by silver nitrate.

**Estimation of Nitrogen**.—(1) Nitrogen as nitrates or nitrites ; (2) nitrogen as ammonia ; (3) nitrogen inert (humus). Place 100 c.c. mercury in a 500-c.c. flask, and cover it with a thin layer of paraffin. Add in small quantities at a time, and with the greatest care, 6·75 grammes sodium. The resulting amalgam will contain 0·5 per cent. Na, and is stored under the paraffin layer.

Triturate 50 grammes of the air-dried soil in a mortar with 200 c.c. ammonia-free distilled water, and convey the thin paste into a litre flask. Add 25 c.c. of soda amalgam, and disseminate the amalgam thoroughly throughout the solution by vigorous shaking. Close the flask, and allow to stand in the cold for twenty-four hours. Add 50 c.c. calcic hydrate, and distil over 100 c.c. into 20 c.c. deci-

normal sulphuric acid. Titrate with decinormal soda, using methyl orange as indicator, or, if the amount is small, Nesslerize.

*Total Nitrogen.*—This is estimated by the Kjeldahl method (*q.v.*). The soil is treated in a pear-shaped flask with 3 grammes benzoic acid and 40 c.c. sulphuric acid, with occasional agitation. One gramme each copper sulphate and mercuric oxide are then added, and after a little, 10 to 20 grammes potassium sulphate. The heating is continued in the usual way, and the nitrogen distilled in the form of ammonia. The amount of nitrogen thus found by using benzoic acid closely corresponds with that obtained by the soda-lime process. Fifteen grammes air-dried soil are mixed with a little sugar and 30 grammes powdered soda-lime, and gently heated in the furnace up to a red heat. The ammonia is absorbed in bulbs containing acid, the contents of which are subsequently distilled with excess of soda by Kjeldahl's process.

• **Analysis of Limestone, Marl, Incrustations, etc.**—The chief constituents of limestone are carbonates of lime and magnesia; other lesser constituents are silica, ferric oxide, iron pyrites, alumina, alkalies, and organic matter.

*Moisture and Organic Matter and Combined Water.*—These are estimated by drying, and igniting at a low red heat.

*Silica.*—Dissolve 1 gramme in dilute hydrochloric acid, evaporate to dryness, bake, and boil up the residue with a fresh addition of dilute acid; wash well with hot water, adding the washings to the filtrate; dry, ignite, and weigh as silica.

*Ferric Oxide and Alumina.*—These are precipitated, together with ammonia, after oxidation with nitric acid, in the filtrate from the silica, washed, dried, ignited, and weighed as such; or they may be separately estimated as under soils.

*Lime.*—To the filtrate from the iron and alumina add ammonium oxalate in excess, then a little ammonia, and boil. Allow to settle, filter, wash well, dry, ignite, and weigh as CaO.

*Magnesia.*—Evaporate the filtrate and washings from the lime to small bulk, cool, acidify with hydrochloric acid, add sodium phosphate solution in excess, as well as ammonia; stir well, and allow to stand for twelve hours; filter, wash with cold ammonia water, dry, and ignite at a white heat. Weigh as magnesium pyrophosphate, which weight, multiplied by 0·36203, equals magnesia (MgO).

*Alkalies.*—Thoroughly mix 1 gramme of the sample with 1 gramme ammonium chloride, and heat at a bright red heat for an hour in a platinum capsule. Boil up the capsule and contents in about 200 c.c. water, and filter. Add slight excess ammonium carbonate, boil, and filter. Evaporate to dryness, heat to dull redness, redissolve in 20 c.c. water, add ammonium oxalate and ammonia in slight excess, boil, and filter. Acidify filtrate with hydrochloric acid, and evaporate the alkaline chlorides to dryness; ignite at a dull red heat; weigh. Dissolve the chlorides in a little water, add platinic chloride in excess, and evaporate to dryness. Treat with alcohol, filter, wash, and dry contents in a tared filter. Weigh as potassium platinic chloride, which, when multiplied by the factor 0·19307, equals potassium oxide ( $K_2O$ ). Obtain the sodium by difference, by deducting the potassium as chloride from the weight of the mixed chlorides; this figure, multiplied by the factor 0·53083, equals sodium oxide ( $Na_2O$ ).

*Sulphates.*—Dissolve 5 grammes of the finely powdered sample in 30 c.c. nitro-hydrochloric acid (1 : 3), evaporate to dryness, and bake. Digest the mass with hydrochloric acid, dilute, filter, and wash. Add to the filtrate, which should not exceed 200 c.c., a slight excess of barium chloride; boil for about twenty minutes, allow to settle for twelve hours, filter, wash with hot water till free from chlorides, dry, ignite, and weigh as usual as barium sulphate.

*Carbonic Acid.*—Determine this by difference in the usual way, using either a Schrotter apparatus or a series of U-tubes.

## CHAPTER VI

### PHOSPHATIC MANURES

**Sampling.**—Dry samples should be ground or chopped, and passed through a sieve having circular perforations 1 millimetre in diameter. Moist samples, too pasty for sieving, should simply be carefully mixed up with the aid of a flexible steel spatula. All samples should be preserved in wide-mouthed bottles for at least three or four months.

**Estimation of Total Phosphoric Acid.**—Take 2 grammes of the sample in a beaker covered with a clock-glass, add about 5 c.c. water, and then, cautiously, a small quantity of hydrochloric acid. Boil to expel all carbon dioxide. Add 20 c.c. aqua regia, and digest on the sand-bath till all but the silica and insoluble matter has dissolved. Filter, wash the precipitate with hot water, and make the cold filtrate up to 200 c.c. Take of this for each estimation 50 c.c., add 50 c.c. molybdate solution, and allow to stand on the top of the water-oven (at a temperature of about 65° C.) for at least one hour. Filter, washing the precipitate by decantation with ammonium nitrate solution. Dissolve the precipitate in ammonia-water, and add excess of magnesia mixture, with constant stirring. Add a further 30 c.c. ammonia (the solution will now amount to about 200 c.c.). Stir every fifteen minutes for two hours, and then allow to settle, or simply stir vigorously, and allow to settle overnight. Filter, wash well with ammonia-water. Dry, ignite at first very gently, and finally at a white heat in a platinum capsule.

If the precipitate is small, it may be placed in the capsule in the wet condition, and ignited without being first dried.

Molybdc solution \*.. . . Molybdc acid, 200 grammes.  
Ammonia (specific gravity, 880), 400 c.c.  
Nitric acid (specific gravity, 1·20), 1,250 c.c.  
Allow to settle, and filter off any precipitate.

Magnesia mixture .. .	Magnesium chloride, 110 grammes. Ammonium chloride, 280 grammes. Ammonia, 700 c.c. Make up to 2,000 c.c. with distilled water; allow to stand several days before use, and decant off from any precipitate.
Ammonia wash-water ..	Ammonia (specific gravity, 880), 100 c.c. Distilled water, 600 c.c.

**Moisture and Organic Matter.**—Take 2 grammes in a tared platinum capsule, dry in oven at 105° C. till weight is constant, ignite over a Bunsen burner, and finish over the blow-pipe or in the muffle for a few minutes. Repeat this ignition until the weight is constant.

**Estimation of Carbon Dioxide.**—Take 2 grammes, and estimate the loss in weight in a Schrotter apparatus, using hydrochloric acid.

**Estimation of Insoluble Matter.**—Take 2 to 5 grammes, and digest with aqua regia on the sand-bath. Filter, wash with hot water. Dry, and ignite residue. Calculate as insoluble matter. The total phosphate may be estimated in this filtrate and washings. The ignited residue is fused with fusion mixture, dissolved in dilute hydrochloric acid, filtered, washed with water, the precipitate ignited and weighed as silica ( $\text{SiO}_2$ ).

**Estimation of Iron and Alumina.**—Take 50 c.c. of the solution in aqua regia, add ammonia in decided excess, redissolve in hydrochloric acid, again render alkaline with ammonia. Add 50 c.c. concentrated acetic acid, stir well, allow to cool and settle. Filter, wash with hot water. Dissolve the precipitate in hot dilute hydrochloric acid, make alkaline with ammonia, and again reprecipitate with acetic acid. Filter, wash precipitate with hot water, dry, ignite, and weigh the combined phosphates of iron and alumina. Redissolve the ignited precipitate in hydrochloric acid, make up to 100 c.c. with water, and estimate the iron by titration as usual. One-half of the ignited precipitate may, with convenience, be assumed as equal to the combined weights of iron and aluminium as oxides.

The aluminium may be estimated by difference, the amount of phosphoric acid having been ascertained.

**Estimation of Lime and Magnesia.**—To the combined filtrate and washings from the iron and alumina estimation an excess of

ammonium oxalate is added, and the solution boiled gently for two or three minutes, allowed to settle, filtered, washed with hot distilled water, dried, ignited, and weighed as CaO. To the filtrate and washings from the estimation of the lime add ammonia in very slight excess, cool well, and then add sufficient sodium phosphate solution to precipitate the magnesia. Stir frequently, allow to stand overnight, filter, wash with ammonia water, dry, ignite, first gently and ultimately at a white heat; weigh as magnesium pyrophosphate.

**Estimation of Sulphates.**—A suitable portion of the original solution may be taken, and the sulphuric acid precipitated in the usual manner with barium chloride.

### Technical Determination of Phosphoric Acid.

#### *List of Reagents employed.*

Molybdate solution .. .	Pure molybdic acid, 110 grammes, dissolved in ammonia (specific gravity, 0·90); add water up to 1,000 c.c.; pour this solution into a litre of nitric acid (specific gravity, 1·2). Allow to stand a few days, then filter for use.
Concentrated ammonium nitrate solution .. .	Pure ammonium nitrate, 750 grammes. Water up to 1,000 c.c.
Magnesia mixture .. .	Magnesium chloride, 55 grammes. Ammonium chloride, 70 grammes. Ammonia (specific gravity, 0·90), 130 c.c. Dissolve and make up to a litre with water.
2·5 per cent. ammonia ..	Ammonia (specific gravity, 0·90), 100 c.c. Water up to a litre.
Joulie's citrate solution ..	Citric acid, 400 grammes, dissolved in ammonia (specific gravity, 0·90), and diluted to a litre.
Wagner's citrate solution ..	Citric acid, 150 grammes, exactly neutralized with ammonia; then add 10 grammes citric acid; dilute with water to a litre.
Sodic acetate solution ..	Crystalline sodium acetate, 100 grammes, dissolved in water; add 100 c.c. acetic acid, and make up to a litre with water.
Calcium phosphate solution ..	Dissolve about 10 grammes pure dry tribasic calcic phosphate in nitric acid, and make up to a litre with water; estimate the phosphoric acid in this gravimetrically with molybdate, and mark the value on the flask.

**Titrated uranium solution.**. Dissolve 250 grammes uranium nitrate in water; add 25 grammes sodium acetate, dilute to 7,000 c.c. One c.c. of this solution equals 0.005 grammie  $P_2O_5$ . To determine its exact strength, proceed as described under volumetric analysis, using 25 c.c. of the above S.S. calcic phosphate.

### Estimation of Phosphoric Acid in Phosphates and Superphosphates.

**Total Phosphoric Acid.**—Take 5 grammes, cover with water, add 30 c.c. hydrochloric acid, and digest on the sand-bath till all has nearly evaporated, and the contents exhibit a characteristic siliceous, jelly-like appearance. Cool, add hot water, agitate. Make up to 500 c.c., and filter off 50 c.c.; to this filtrate add 15 c.c. Joulie's solution. Stir well at intervals, allow to stand for some hours, preferably overnight. Filter. Wash with ammonia-water (1 : 6). Dry, ignite, and weigh as magnesium pyrophosphate.

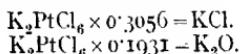
**Citrate Soluble Phosphoric Acid.**—Place 1 grammie in a 100-c.c. flask, and add 30 c.c. Wagner's citrate solution and 30 c.c. water; warm the flask to about  $40^{\circ}\text{C}$ ., and agitate well for an hour. Make up contents of the flask to 100 c.c., and filter off 50 c.c. In this filtrate precipitate the phosphates as described above with magnesia. Dissolve the precipitate of magnesium ammonium phosphate in a small quantity of nitric acid, neutralize with ammonia, add acetic acid in excess, and finally titrate the phosphoric acid with S.S. uranium acetate.

**Estimation of Phosphoric Acid in Superphosphates (Water Soluble).**—Take 20 grammes of the superphosphate, grind up in a mortar with, first, 150 c.c. water, pouring the clear solution off through a filter into a litre flask; then repeat this treatment with 100 c.c. and 50 c.c. water in a beaker. Filter, transferring all to the filter. Wash well with water. Cool filtrate, and make it up to 1,000 c.c. Shake up well. Take an aliquot part, and precipitate the phosphoric acid in a beaker with magnesia mixture. Wash, ignite, and weigh the pyrophosphate in the usual manner; dissolve it in a small quantity of nitric acid, render alkaline with ammonia, add sodic acetate, and titrate as usual with S.S. uranium acetate.

### Estimation of Potash.

Of the potassium salt about 0·7 gramme is taken for analysis. Take 35 grammes of "muriate," dissolve in a small quantity of water, filter into a 500-c.c. flask, and make up to the mark. Use 10 c.c. (equal to 0·7 gramme). Take 17·5 grammes of sulphate, proceed as before mentioned, and use 20 c.c. Place the aliquot part in a porcelain basin, dilute to 25 c.c., and slightly acidify with hydrochloric acid. Add about 30 c.c. platinic chloride (about 10 per cent.), and evaporate over a water-bath to dryness. Drench with a few c.c. of platinic chloride, stir well, and allow to settle for some time. Filter through a small filter, washing out the basin with a few drops of the platinic chloride. Then wash dish, filter, and contents with a minimum quantity of 95 per cent. alcohol, and dry at 100° C. Transfer the precipitate to a tared porcelain crucible, complete the drying, and weigh. Ignite the filter with any adhering precipitate on the crucible lid, and multiply the residue by 1·41, after deducting the weight of the ash.

Sulphates and nitrates do not require removal by this process, nor does magnesia, if present, interfere.



### Rapid Control Method for Potash Salts (Albert).

Place the finely ground substance in a litre flask, add about 400 c.c. water and 3 c.c. hydrochloric acid ; add barium chloride drop by drop as long as a precipitate is produced. Cool, make up to mark, shake, filter through a dry filter. Evaporate an aliquot part to almost dryness, and treat the mass with alcohol. Filter through a tared filter, and wash well with alcohol. Dry filter in oven till constant in weight.

Kainit or Carnallite	20 grammes in 1 litre ; of that 50 c.c. with 40 c.c. $PtCl_4$ . Weight of $K_2PtCl_6 \times 19\cdot3$ = per cent. $K_2O$ .
Sulphate of potash . .	15 grammes in 1 litre ; of that 20 c.c. with 15 c.c. $PtCl_4$ . Weight of $PtK_2Cl_6 \times 64\cdot33$ = per cent. $K_2O$ .
Chloride of potash . .	10 grammes in 1 litre ; of that 25 c.c. with 15 c.c. $PtCl_4$ . Weight of $K_2PtCl_6 \times 77\cdot2$ = per cent. $K_2O$ .

### Recovery of Platinum from Platinum Waste, Precipitates, etc.

The residues and filtrates, etc., are boiled with excess of soda solution in a porcelain basin. Methylated spirit is added, and the liquid again boiled, when reduction takes place, the completion of which is observed when the platinum black falls to the bottom. Decant off the clear liquid, and wash the residue first with a little dilute sulphuric acid, and then with water, till the washings are clear. Add to the residue about twice its bulk of hydrochloric acid, and boil ; dilute, filter, and wash with hot water. Dry and ignite in a porcelain crucible, cool, and weigh. Boil the dried residue with pure nitric acid, and wash till free from acid. Dissolve in aqua regia ( $3\text{HCl} : 1\text{HNO}_3$ ), and evaporate the solution twice with dilute hydrochloric acid and once with water. Take up in as much water, as will yield a solution containing 6 to 10 per cent. metallic platinum.

## CHAPTER VII

### ESTIMATION OF NITROGEN

#### The Soda-Lime Process (Will and Varrentrap).

This process consists in decomposing the nitrogenous substance, intimately mixed with soda-lime, at a red heat, thereby generating ammonia gas, which is absorbed by a known quantity of S.S. sulphuric acid, with subsequent estimation of the amount of acid thus neutralized by titration with S.S. soda hydrate.

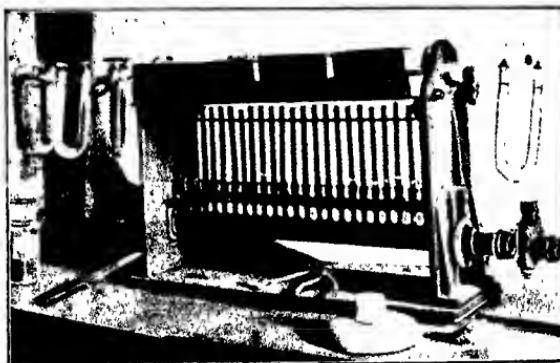


FIG. 18.—COMBUSTION FURNACE.

The combustion is carried out in a hard glass or iron tube,  $\frac{1}{2}$  inch internal diameter, sealed at one end, and connected with a suitable absorption vessel.

The quantity operated upon may vary from 0.3 grammes, as in the case of pure urea, to 2 grammes in the case of nitrogenous manures.

The lower or closed end of the tube is loosely packed with a little recently ignited asbestos, and then a little fine soda-lime is introduced. The substance, having been accurately weighed out, is placed in a mortar, and thoroughly mixed with a small quantity of the soda-lime, the mortar being rinsed out with fresh granulated soda-lime. The mixed sample is then transferred, together with

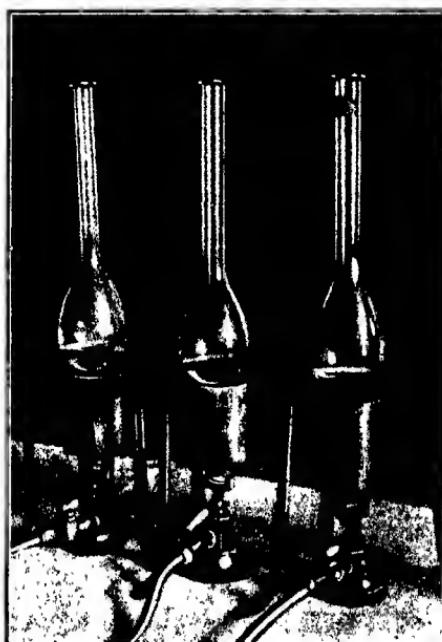


FIG. 19.—ESTIMATION OF NITROGEN: KJELDAHL PROCESS  
(DIGESTION FLASKS).

the rinsings, into the tube by means of a funnel, and the tube filled up to within 4 inches of the open end with soda-lime, and covered with a slightly pressed 1-inch layer of asbestos in the form of a plug. Now connect on the Will and Varrentrap bulb by means of a tightly fitting cork, and place the tube in its place in the combustion furnace, covering it up with the tiles. Next light the

burner next to the open end, and wait until all the gas has been evolved. As soon as this is effected proceed likewise with each burner in turn until the whole tube is heated. Continue the heating at a red heat until all bubbling of gas in the acid has ceased. Disconnect the bulb, and transfer the acid to a beaker or flask, rinsing it out two or three times with distilled water. Add a few drops of methyl orange, and titrate back to neutralization with quintinormal sodium hydrate.

If seminormal sulphuric was used, multiply the number of c.c. of quintinormal soda used in neutralizing the remaining free acid by 2·5, and subtract this from the number of c.c. of seminormal acid originally taken, when the remainder will represent the amount neutralized by the ammonia. Twenty c.c. is the usual amount of seminormal acid employed in this estimation.

From this the percentage of ammonia and nitrogen is obtained by reference to the tables given.

### The Sulphuric Acid or Kjeldahl-Gunning Process.

This process consists in decomposing the nitrogenous substance by nitrogen-free sulphuric acid in presence of potassium sulphate, whereby the nitrogen in the form of ammonia is converted into sulphate, and subsequently distilled with excess of alkali, and absorbed by a known quantity of standard sulphuric acid.

It is necessary that a blank experiment be carried out whenever fresh material is used. The acid should be nitrogen-free. A strong solution of sodium hydrate—say 360 grammes per litre—is prepared, and the necessary excess quantity ascertained by experiment, this quantity being strictly adhered to in all subsequent estimations. The potassium sulphate should be pure and anhydrous.

*Process.*—Weigh out accurately 1 to 2 grammes of the sample, and place it in a long-necked, pear-shaped Jena glass flask. These flasks are stocked for this purpose. Add 20 c.c. of the sulphuric acid, and heat gently over an Argand burner in the funie cupboard till all violent action has ceased. Rapidly transfer by means of a paper funnel 10 grammes of the potassium sulphate, and continue the heating until the contents have assumed a pale straw tint, or, if possible, a white colour.

Cool, add a little water, and transfer the solution to the distillation flask, rinsing out the oxidation vessel several times. The contents of the distillation flask should now measure about 150 c.c. Measure out the requisite quantity of alkali, and quickly run it

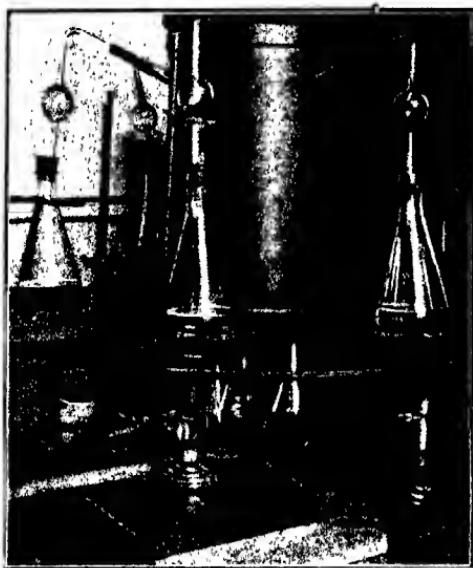


FIG. 20.—ESTIMATION OF AMMONIA : KJELDAHL PROCESS.

into the flask. Boil for about half an hour, or until all the ammonia has been driven over into the receiver. This being accomplished, next titrate the contents of the receiving flask as described in the soda-lime method.

#### Nitrometer Method for Nitrates.

This is described under Water Analysis.

## CHAPTER VIII

### ANALYSIS OF FEEDING-STUFFS

FOOD-STUFFS of a vegetable character may be examined qualitatively, as to their condition and purity, or quantitatively, the usual quantitative course consisting in the determination of moisture, ash, ether extract, proteids, total carbohydrates, and crude fibre.

#### Quantitative Analysis.

**Moisture.**—From 2 to 3 grammes of the finely ground sample are spread out on a tared platinum or porcelain dish or watch-glass, and dried for five hours in the water-oven.

**Ash.**—The above dried residue is gently ignited to whiteness at a low red heat, cooled, and weighed.

**Silica.**—The ash is transferred to a small beaker with the aid of a little dilute hydrochloric acid, digested on the water-bath, filtered, and well washed with hot water. The wet filter and contents is then ignited directly, cooled, and weighed.

**Ether Extract (Oil).**—Place 2 to 3 grammes of the dried sample in a Soxhlet thimble, and extract with anhydrous alcohol-free ether for from two to six hours, depending upon the nature of the sample—oil-cakes, for example, usually require two hours.

**Total Carbohydrates.**—These are generally approximately obtained by difference.

**Crude (or Woody) Fibre.**—Extract the residue from the determination of the ether extract in a 500-c.c. beaker with 50 c.c. 5 per cent. sulphuric acid and 75 c.c. water by boiling gently for half an hour. Allow to settle, after having filled up with cold water, and decant the supernatant liquid through a piece of clean

muslin, washing back any particles that may have come over on to the cloth.

Add 50 c.c. 5 per cent. KOH and 75 c.c. water, and again boil for half an hour, making the volume up as before with cold water, and filter through a tared filter. Wash three times with hot water, once with dilute hydrochloric acid, once with water, and finally once each with alcohol and ether. Dry and weigh, and deduct the weight of the ignited fibre and filter ash from the weight thus obtained ; the loss in weight is the crude fibre.

**Albuminoids.**—Determine the nitrogen on 1 to 2 grammes of the sample either by the soda-lime or Kjeldahl process, multiply the percentage thus formed by the factor 6·25, and thus obtain the percentage of albuminoids. The factor 6·25 is a general or average one, various cereals having their own special numbers.

#### EXAMINATION AND ESTIMATION OF THE CARBOHYDRATES.

**Starch** ( $C_6H_{10}O_5$ )<sub>n</sub>.—Starch occurs in plants in various proportions at different stages of their development. Starch is insoluble in cold water, alcohol, or ether ; soluble in hot water, with change ; and is hydrolyzed by boiling with dilute acids first into dextrin and maltose, and finally into dextrose.

Starch may be detected by testing its cold solution with a few drops of iodine, when a characteristic blue colour will be produced if it is present. When present in minute quantities, this reaction may be, with convenience, carried out under the microscope.

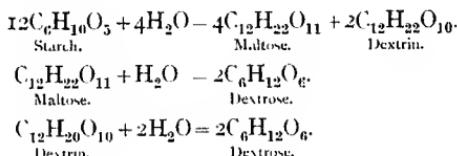
Starch granules differ in their form, size, and distribution when viewed through the microscope, either with normal or polarized light, according to their origin.

The student should therefore accustom himself to the morphological identification of the various starches by first preparing a series of slides, mounted either in water or glycerin, from samples of known purity, including, amongst others, wheat, barley, rye, corn, oat, pea, bean, rice, potato, and arrowroot.

**ESTIMATION OF STARCH**—*Direct Acid Conversion.*—This method is fairly accurate in cases where there are very little insoluble carbohydrates (hemicellulose, pentosans), as such bodies are also converted into sugar by the action of the acid.

Treat 2 to 3 grammes of the fat-free sample with 20 c.c. hydrochloric acid and 200 c.c. water by boiling under a reflex condenser for three hours. Cool, neutralize with sodium hydrate, filter if necessary, and estimate the dextrose either by Fehling's solution or in the polarimeter. Calculate the dextrose into starch by the factor 0·9.

*Diastase Conversion.*—This method is more accurate than the above, and the hemicellulose is unaltered, the reaction being expressed according to the equation—



Macerate 20 grammes powdered malt with 100 c.c. water for several hours, filter, destroy further growth by adding a few drops of chloroform, and estimate the dextrose in this extract obtained by treating a known quantity as above with acid.

Heat 2 to 3 grammes of the fat-free sample in a 250-c.c. flask with 50 c.c. water until the starch gelatinizes. Cool to 55° C., add 40 c.c. of the above malt extract, and allow to stand at that temperature until all the starch is converted. This may be determined by testing a drop of the solution with iodine. Dilute to 250 c.c., filter off 200 c.c., to which add 20 c.c. hydrochloric acid, and heat on the water-bath under a reflex condenser for three hours. Cool, neutralize with sodium hydrate, and estimate the dextrose as in the "acid conversion" process, deducting the amount of dextrose present in the quantity of malt extract used.

**Cellulose.**—This is insoluble in water, alcohol, or ether, but readily soluble in Schweitzer's reagent (ammoniacal cupric hydroxide). Cellulose becomes blue in colour when treated with sulphuric acid and iodine in potassic iodide, and violet with zinc chloriodide.

**Pentosans.**—These are insoluble carbohydrates, or "hemicellulose," capable of direct acid conversion into pentose sugars. The hexosans are similarly hydrolyzed into hexose sugars (mannose,

galactose, etc.). These compounds may be conveniently estimated by boiling the residue from the malt extract starch determination with 100 c.c. 1 per cent. hydrochloric acid, filtering, washing with water, and estimating the sugar in the filtrate in the usual manner. The results may be expressed in terms of their principal sugar—viz., xylose.

$$\text{Xylose} \times 0.88 = \text{xylan.}$$

$$10 \text{ c.c. Fehling solution} = 0.046 \text{ grammes xylose.}$$

**Cane-Sugar.**—Extract 50 to 100 grammes of the sample with 300 to 500 c.c. 95 per cent. alcohol under a reflex condenser for three hours, filter, evaporate to small bulk, and filter off from any oily matter after dilution with water. Estimate the sugar as cane-sugar (sucrose) in a portion of this filtrate with Fehling's solution after inversion with hydrochloric acid. Determine the reducing sugar, dextrose, if present, in another portion of the same filtrate.

**Dextrin.**—Digest the above residue from the sugar determination with 300 to 500 c.c. cold water for twenty-four hours with frequent shaking. Filter, invert with acid, and determine the sugar thus formed with Fehling's solution.

**Albuminoids.**—See chapter on Proteids, etc.

**Gluten in Flour.**—Knead to a paste 30 grammes of the sample with water, and strain off the starch through a fine muslin or silk bag. Wash thoroughly free from starch under water, compress into a ball, wipe dry, and weigh.

#### GRASS AND HAY.

**Moisture.**—Counterpoise two large filter-papers, the one against the other, on a large precision balance, sensitive to 0.005 gramme. Weigh out 20 grammes hay or 50 grammes grass, and dry with both papers in the water-oven till no further loss in weight is found. The dried sample is then bottled and termed "dry matter."

**Woody Fibre.**—This is estimated as already described under feeding-stuffs. Any green colouring matter retained in the fibre after extraction may be removed by soaking for an hour, first with alcohol and then with ether. Deduct the weight of the ash from that of the dried fibre.

**Crude Fibre.**—Macerate a suitable weighed quantity in a large beaker with cold water, first for twenty-four hours, secondly for three hours, and lastly for one hour, carefully decanting off the liquid, and adding fresh water each time. Then boil four times with four volumes of water. Extract the oil and colouring matters with alcohol and ether as above described, dry and weigh. Store this crude fibre in a bottle.

"DRY MATTER"—*Total Nitrogen*.—Estimate this in 2 grammes of the thoroughly dry substance by the Kjeldahl process.

*Total Albuminoids*.—Macerate 2 grammes of the matter with a 4 per cent. aqueous solution of carbolic acid, containing a drop or so of metaphosphoric acid, for twenty-four hours. Decant through a tared filter, and digest at boiling-point with a fresh quantity of the carbolic acid, filter, wash with the carbolic acid solution, and dry.

- In this way the amides are eliminated, and the albuminoids are coagulated and retained in the filter.

Estimate the nitrogen in the dried residue, and express as albuminoids ( $= N \times 6.25$ ).

*Total Ash*.—Carefully ignite 2 grammes of the dry matter in a platinum capsule.

"CRUDE FIBRE"—*Insoluble Albuminoids*.—Estimate these on 2 grammes as in the "dry matter."

*Insoluble Ash*.—Estimate on 2 grammes as in the "dry matter."

In this way the following data have now been obtained :

Sample . . . . . " Dry matter " . . . . . " Crude fibre " . . . . .	Moisture, per cent. Woody fibre, per cent. Crude fibre, per cent. Total nitrogen, per cent. Albuminoid nitrogen, per cent. Total ash, per cent. Insoluble albuminoids, per cent. Insoluble ash, per cent.
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Calculate these percentages to percentages of the sample "as received." Then—

$$\text{Total nitrogen} - \text{albuminoid nitrogen} = \text{amide nitrogen}.$$

$$\text{Total albuminoids} - \text{insoluble albuminoids} = \text{soluble albuminoids}.$$

$$\text{Total ash} - \text{insoluble ash} = \text{soluble ash}.$$

Crude fibre—[insoluble albuminoids + woody fibre + insoluble ash]  
= digestible fibre.

Ascertain the colouring matter (chlorophyll), etc., by difference.

#### SILAGE.

e

Determine the constituents as in grass or hay, and in addition estimate the acidity in the combined extractions and washings obtained in estimating the crude fibre. For this purpose the solution may be made up to fixed volume, and an aliquot portion taken for titration with decinormal alkali, using phenolphthalein as an outside indicator. This provides the total acidity. Another aliquot part of the solution is now boiled till free from volatile (acetic) acid, titrated in a similar manner, and expressed in terms of acetic acid.

The difference in the amount of alkali used in determining the total and volatile acidity is due to the fixed acids, and is expressed as lactic acid.

#### ROOTS (VARIOUS).

The principal constituents determined in such plants as swedes, mangels, and turnips are water, cellulose, pectose, gum, sugar, albuminoids, and mineral matters, with perhaps the fatty, colouring, and other substances.

As the amount of water present is very large—from 80 to 90 per cent.—much, if not all, of the soluble matter will be in solution. The analysis is therefore resolved into two main divisions—viz., the analysis of the juice and that of the residual insoluble portion or fibre.

**Preparation of Sample and Estimation of Moisture.**—Remove the leaves and adhering soil from several average roots, and slice each, from top to bottom, into six or eight pieces. Take one thin slice from each root, and weigh out a 100-gramme average sample of the whole. Dry gradually till the most of the moisture is driven off, and then in the water-oven until no further loss is sustained. Store the dried sample in a bottle.

**Estimation of Crude Fibre.**—Grate down to a fine pulp a fresh section of each of the roots, and of this weigh out 500 to 1,000 grammes. Place in a linen bag, compress in a meat or cheese

press, and receive the juice into stoppered flasks. Weigh the residual fibre, and estimate the crude fibre on 25 to 50 grammes, and the woody fibre on 5 to 10 grammes of this pulp by the methods described under Grass and Hay, and estimate the ash and nitrogen in the former. The analysis of the juice must be carried out as soon as possible, as it rapidly decomposes and alters in its composition. The liquid should be made up to a fixed volume, and its specific gravity ascertained. The following constituents are then determined : glucose, cane-sugar, soluble albuminoids, total solids, and soluble ash.

**Glucose.**—Pipette 10 c.c. into a 100-c.c. flask, add a little water and 10 c.c. of a saturated solution of lead acetate, make up to the mark with water, mix thoroughly, and allow to settle for a few hours. Filter, and estimate the glucose with Fehling's solution in the manner described in the chapter upon Sugar Analysis. Should an excessive quantity of lead be required for the purposes of clarification, it must be removed by carefully adding the requisite amount of a solution of alum.

**Cane-Sugar.**—This is ascertained by estimating the total sugar, and subtracting from it the number of c.c. of Fehling's solution required in the estimation of the glucose.

Pipette 50 c.c. of the saccharine liquid into a beaker, add 10 c.c. dilute acid, and invert the sucrose. Filter, neutralize with alkali, and make up to 100 c.c. with water. Cool, and titrate with Fehling as usual.

**Soluble Albuminoids.**—Pipette 50 c.c. of the juice into a beaker, add a few drops of lactic acid, mix, and allow to stand for twenty-four hours, in order to coagulate all the albuminoids. Filter, wash with hot water, dry in the water-oven, estimate the nitrogen in the dried mass by the Kjeldahl process, and calculate to albuminoids by multiplying by the factor 6.25.

**Total Solids.**—Concentrate 50 c.c. of the juice in an evaporating-basin upon the water-bath, breaking up any scum with a platinum rod. Dry thoroughly in the water-oven, and weigh until constant in weight.

**Soluble Ash.**—Ignite the dry solids in a platinum capsule, and estimate the soluble portion as has been already described under other foods.

The following data will now have been obtained :

Sample	..	..	..	Pulp.
Pulp	..	..	..	Water.
Crude fibre	..	..	..	Crude and woody fibre.
Juice	..	..	..	Nitrogen (insoluble albuminoids).
Dried pulp	..	..	..	Ash.
				Glucose.
				Cane-sugar.
				Soluble albuminoids.
				Ash.
				Nitrogen.
				Ash.

Reduce all percentages to percentage of sample, as in the case of grass and hay. Then—

Crude fibre—[woody fibre + insoluble albuminoids + ash] = digestible fibre.

**Pectose, Pectin, and Peptic Acid in Roots.**—These are generally estimated and expressed as pectic acid.

Macerate a suitable weighed quantity of the sample with alcohol for twelve hours, strain through muslin, and further treat with a mixture of 3 parts alcohol to 1 part hydrochloric acid, filter, and wash with alcohol till free from acid. Digest with 150 c.c. of an alcoholic solution containing 1 gramme potassic carbonate for thirty minutes at 75° C. under a reflex condenser. Filter and wash with alcohol, make the volume of the filtrate and washings up to 700 to 800 c.c. in a 1,000-c.c. flask, add 2 grammes ammonium oxalate, and heat for two hours at a temperature of 35° C. Filter, wash, and make the cooled solution up to 1,000 c.c.

The pectose, pectin, and peptic acid, now existing as ammonium pectate, is precipitated in an aliquot portion of the solution (100 c.c. or thereabouts) with a slight excess of a solution of calcium acetate. Filter off the calcium pectate and oxalate, wash first with water, and then with alcohol, dry, weigh, ignite, and weigh as calcium oxide.

From the amount of ammonium oxalate used that of the calcium oxalate can then be calculated.

Let  $a$  = amount of calcium oxalate,

$b$  = weight of CaO, less that contained from the oxalate,

$c$  = weight of the precipitate of calcium pectate and oxalate from the aliquot portion, dried in the water-oven,

Then  $x$  = the weight of pectic acid,  
 $= c - (a + b)$ .

## CHAPTER IX

### ANALYSIS OF DAIRY PRODUCTS

#### Milk.

MILK is the mammary secretion of female mammals, and contains fat, casein, albumin, sugar (lactose), salts, and water.

Fresh human milk, as well as that from the herbivora, has an alkaline reaction, the milk of the carnivora being acid. Cow's milk usually exhibits an amphoteric reaction, or, in other words, appears to be acid and alkaline at one and the same time.

Milk fat, like other animal fats, is a glyceride, and yields glycerol and a fatty acid upon saponification, the chief saponification acid being butyric acid ( $C_4H_8O_2$ ).

The milk proteids consist of casein, albumin, and peptones. These are all precipitated by acids and various metallic salts, and are gradually resolved into less complex substances on decomposition.

Milk-sugar, or lactose ( $C_{12}H_{22}O_{11}$ ), has only been obtained as yet from human milk and from that of the herbivora. Anhydrous milk-sugar is dextro-rotatory ( $a_{1D}55^{\circ}27'$ ). It reduces alkaline solutions of cupric salts, but less readily than grape-sugar, and is hydrolyzed by boiling with dilute mineral acids yielding the glucoses—dextrose and galactose.

The mineral constituents of milk vary somewhat, ranging from the lowest in the human and mare to that of the cow, the sheep being highest.

Four analyses of the ash of cow's milk are published by Weber and Haidlen :

	Minimum.	Maximum.	Mean.		Minimum.	Maximum.	Mean.
Potash ..	17.09	33.25	24.67	Phosphoric			
Soda ..	8.60	11.18	9.70	anhydride	27.04	29.13	28.45
Lime ..	17.31	27.55	22.00	Sulphuric			
Magnesia ..	1.90	4.10	3.05	anhydride	—	—	0.30
Ferric oxide ..	0.33	0.76	0.53	Chlorine ..	9.87	16.96	14.28

The ratio that the ash of milk bears to the total solids is from 7·8 to 9·4 : 100.

#### ANALYTICAL PROCESSES.

**Specific Gravity.**—This is obtained by means of the lactometer, which is simply a hydrometer graduated from 15° C. (specific gravity, 1·015) to 45° C. (specific gravity, 1·045). The sample is poured into a hydrometer jar, and its temperature adjusted to 15·5° C., when the lactometer is gently lowered into it, and the reading is observed from the top of the meniscus. The specific gravity of pure milk varies from 1·030 to 1·040. The addition of water lowers the specific gravity, and the removal of fat increases it.

**Total Solids.**—From 5 to 10 grammes are carefully weighed out, or, if the specific gravity is known, measured out, into a flat porcelain evaporating dish about 2½ inches in diameter. The dish must have been previously tared, and the milk should form a thin even layer on the bottom of the dish. The dish and contents are next heated in the water-oven for three hours and weighed, and again heated for subsequent periods of half an hour until the weight becomes constant. The final weight of the dish and dried contents minus the weight of the dish gives the weight of the total solids.

Richmond has based a formula upon a large number of analyses, whereby, given any two of the following, the third may be found —viz., the specific gravity, total solids, or fat. A handy sliding scale for this purpose is sold by most dealers, and suffices to give a rapid and fairly accurate estimation of the fat or other third constituent.

**Ash.**—Platinum dishes may be used for the estimation of the total solids, and the ash thereafter rapidly found by ignition at a dull red heat, care being taken not to volatilize the alkaline salts by too high a temperature. Porcelain dishes may be used with soft Bunsen flames. Nickel dishes are to be avoided.

**Fat.**—*The Werner-Schmidt Process.*—This process is capable of producing very accurate results. Shake the milk thoroughly, introducing if thickened a few pellets of dry clean lead shot, and transfer 10 grammes or 10 c.c. of it to the Werner-Schmidt tube. Add 10 c.c. concentrated hydrochloric acid, cork tightly with a rubber cork, and heat over the water-bath for about ten minutes, with frequent

shaking, until the liquid is of a clear deep brown colour. The tube must not be heated too long; otherwise carameloid bodies are formed, which become intimately mixed with the fat, and prevent its accurate estimation.

After heating, the tube is suddenly and thoroughly cooled in a stream of water, filled to the 50-c.c. graduation with ether, again vigorously shaken, and allowed to stand until the ether layer has completely separated out.

Next measure off rapidly two 10 c.c. portions of the supernatant

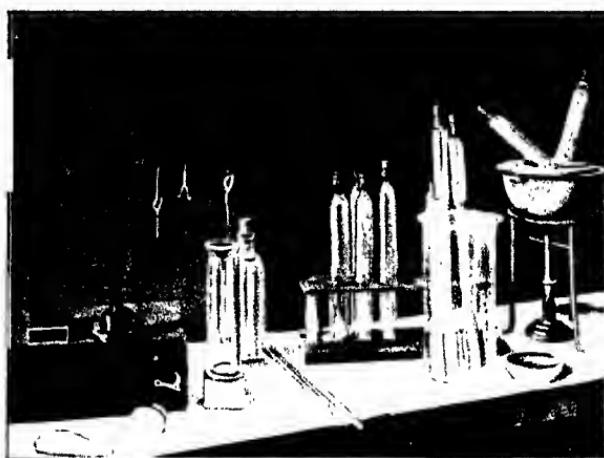


FIG. 21.—MILK ANALYSIS: WERNER-SCHMIDT PROCESS.

etheral solution, after having carefully noted its exact volume, into two tared small beakers, and gently dry them on the top of the water-oven. When free from all traces of ether, cool, and weigh the beakers containing the fat.

*Example—*

Took 10 grammes of the milk.

After extraction the etheral solution measured 27 c.c.

10 c.c. etheral solution gave 0·13 grammes fat.

Therefore 10 c.c. of the milk equals  $0\cdot13 \times 27 \div 10 = 0\cdot351$ .

Therefore the milk contains 3·51 per cent. fat.

An alternative and more accurate method is to transfer almost all the ethereal solution by means of a pipette with small aperture to the tared beaker, and repeat the extraction of the remaining traces of fat by subsequent agitations with fresh quantities of ether until all the fat is removed.

*Adam's Coil Process.*—A weighed or measured quantity of the



FIG. 22.—SOXHLET APPARATUS : ADAM'S COIL PROCESS.

milk is poured upon a strip of C. S. and S. fat-free paper (supplied in boxes of 25 and 100), and is dried by placing in the water-over for some time. The dried paper is now rolled up lightly, and placed in a Soxhlet fat-extraction apparatus, and the fat extracted by pure ether (specific gravity, 0'720). It is very essential that the ether be dry; otherwise proteids are apt to be dissolved. After

allowing the ether to circulate through the coil at least twenty times, the tared flask and ethereal contents are detached, the ether regained by distillation over a water-bath by means of a long Liebig's condenser, and the flask and fat weighed until constant. Care must be taken not to heat the flask and contents at too high a temperature or for too long a period, as oxidation may ensue, and the result be too high in consequence.

*Gerber Process.*—This consists in separating the fat by centrifugal



FIG. 23.—MILK ANALYSIS: LEFFMANN-BEAM PROCESS.

force. Place 10 c.c. sulphuric acid (specific gravity, 1.820 to 1.825) in one of the bottles, add 1 c.c. pure amyl alcohol, and then with great care 11 c.c. of milk sample.

Cork the tube firmly, and rotate for two minutes or so, when the volume of fat is read off to within 0.1 per cent.

*Leffmann-Beam Process.*—This is a centrifugal apparatus, also, capable of holding two, four, eight, or twelve bottles at one time. Fifteen c.c. of the milk are measured into one of the bottles, and 3 c.c. of a mixture of equal parts of amyl alcohol and fuming hydro-

chloric acid added. The solutions are well mixed by rotating the bottle by the hand, and then 9 c.c. sulphuric acid (specific gravity, 1·83) is slowly added with care. The bottle is then placed in the machine and whirled for two minutes. If the milk is poor or skim then three to four minutes will suffice. The fat is read off to within 0·1 per cent.

#### REMARKS ON THE RESULTS OF MILK ANALYSIS.

The average composition of milk from 200,000 analyses by Vieth and Richmond is :

	Per Cent.
Water .. .. . .. .. .	87·10
Fat .. .. . .. .. .	3·90
Milk-sugar .. .. . .. .. .	4·75
Casein .. .. . .. .. .	3·00
Albumin .. .. . .. .. .	0·40
Ash .. .. . .. .. .	0·75

The minimum quantity of solids not fat contained in fresh milk, according to the regulations of the Sale of Foods and Drugs Act, is fixed at 8·5 per cent., and that of fat at 3 per cent., so that, where the solids not fat in the milk are deficient,

$$\text{Added water} = 100 - \frac{s}{8\cdot5} 100,$$

and where  $f_1$  = fat in original milk (3 per cent.), and  $f_2$  = fat in sample,

$$\text{Per cent. fat abstracted} = 100 \frac{f_1 - f_2}{f_1}.$$

The present standard for solids not fat in skinned milk is 9 per cent.

Fat in milk tends to increase in the winter months, attaining its highest point in November. This is largely due, of course, to the nature of the feeding.

**Estimation of Lactose in Milk.**—Milk-sugar is most conveniently determined in the unaltered milk volumetrically by Pavy's solution. To prepare this solution mix 120 c.c. ordinary Fehling's solution with 300 c.c. ammonia (specific gravity, 0·880) and 100 c.c. of 10 per cent. caustic soda. Pavy's solution may also be made up by dissolving 4·157 grammes pure recrystallized cupric sulphate in about 100 c.c. water, and adding to this a solution of 21·6 grammes

pure recrystallized Rochelle salt and 18·4 grammes caustic soda (or 25·8 grammes caustic potash). To the cold mixture 300 c.c. ammonia (specific gravity, 0·880) is added, and the whole is made up to 1,000 c.c.

One hundred c.c. Pavy's solution is equal to 10 c.c. Fehling's solution, or 0·05 gramme glucose.

Pass the nose of a burette through a cork, provided with three holes, into a wide-necked 150 c.c. flask; the other two holes admit an exit-tube for the ammoniacal fumes and a safety-tube. Measure into the flask 50 c.c. of Pavy's solution, add a piece of platinum foil to prevent bumping, and run in the sugar solution from the burette slowly, and with constant agitation. The total destruction of the blue colour marks the end of the reaction, and the end-point is very sharp. From the amount of sugar and Pavy solutions used the percentage of lactose is calculated in the usual manner.

\* A polarimetric method of estimating lactose in milk is to add to 60 c.c. of the sample 10 c.c. of a solution of mercuric nitrate prepared by dissolving mercury in twice its weight of nitric acid (specific gravity, 1·42), and diluting with four volumes of water. The resulting mixture is diluted to 100 c.c., well shaken, and filtered through a dry filter. The clarified sugar solution is then placed in a 400-millimetre observation-tube, and its rotation at once noted. A similar quantity of milk is treated in the same way, but in this case diluted up to 200 c.c. From the rotations thus observed the following data are deduced: viz., the true volume of liquid in the flask, the true percentage of lactose, and the true volume of the precipitate.

Let  $x$  equal the volume of the precipitate.

Let  $y$  equal the correct reading of the polarimeter.

Let  $a$  equal the reading from 100 c.c.

Let  $b$  equal the reading from 200 c.c.

$$1. \quad 200-x : 100-x :: a : b.$$

$$\text{Therefore } x \text{ equals } 100 \cdot \frac{(a-2b)}{(a-b)}.$$

$$2. \quad 100-x : 100 :: y : a.$$

$$\text{Therefore } y \text{ equals } \frac{(ab)}{(a-b)}.$$

## PRACTICAL AGRICULTURAL CHEMISTRY

Therefore the polarization is the quotient obtained by dividing the product of the two readings by their difference.

**Detection of Cane-Sugar or Glucose in Milk.**—Ten c.c. of the sample are treated with 5 grammes of powdered ammonium molybdate and 10 c.c. dilute hydrochloric acid (1 : 10). A blank experiment is at the same time also started with a pure milk. The two tubes are gradually heated on the water-bath up to 80° C. If the sample contains cane-sugar or glucose, it will assume a blue colour.

**Detection of Glycerine in Milk.**—Curdle the milk, evaporate the whey to dryness, and remove all traces of fat with ether. Dissolve the glycerine in a mixture of alcohol and ether, evaporate the filtered solution, and weigh the residual glycerine.

**Detection of Gelatine in Milk.**—Dissolve mercury in twice its weight of concentrated nitric acid, and dilute it twenty times with water (Wiley's solution). Add 10 c.c. of this solution to an equal volume of the sample, shake, add 20 c.c. water, again shake, allow to stand for five minutes, and filter. The solution will be opalescent if much gelatine be present; on the addition of a solution of picric acid a yellow precipitate will indicate the presence of gelatine.

**Detection of Boric Acid in Milk.**—Evaporate 5 or 10 c.c. of the sample to dryness, and slowly char the residue; moisten the ash with a few drops of concentrated sulphuric acid, and then add a little alcohol. Apply a light. The presence of boron compounds will be demonstrated by a greenish coloured flame.

**Estimation of Boric Acid in Milk.**—Make 100 c.c. of the milk distinctly alkaline with caustic soda, evaporate the mixture and char the residue. Extract the charred mass with a little hot water, and add hydrochloric acid drop by drop until only carbon is left undissolved. Add 0.5 to 1 gramme dry calcium chloride. Add a few drops of phenolphthalein, and titrate with a 10 per cent. solution of caustic soda until a pink coloration is just attained. Add 25 c.c. lime-water; make up to 100 c.c., shake well, and filter through a dry filter into a 50-c.c. flask. Titrate 50 c.c. of the filtrate with normal sulphuric acid until the pink colour disappears; add a few drops of methyl orange, and more acid until the yellow changes to pink. Titrate back very carefully with quintinormal

caustic soda until the yellow just reappears. Boil to expel carbon dioxide, cool, add at least 30 per cent. of glycerine, and finally titrate with quintinormal caustic soda.

Each c.c. of soda used in the final titration equals 0.0124 grammes boric acid, or 0.007 grammes  $B_2O_3$ .

**Detection of Formalin in Milk.**—To 10 c.c. of the sample add 1 drop of ferric chloride and dilute the milk to about 30 c.c.; pour down the side of the tube, cautiously, a little concentrated sulphuric acid, so as to form a layer at the bottom. A violet ring at the junction of the liquids will appear if formaldehyde is present.

**Detection of Salicylic Acid in Milk.**—Extract a little of the milk with ether, after solidifying with hydrochloric acid, and evaporate the ethereal solution to dryness. A violet coloration on the addition of a solution of ferric chloride will reveal the presence of salicylic acid.

**Detection of Raw Milk in Boiled or Sterilized Milk.**—Warm the sample gently with a few drops of fresh oil of turpentine, and add a few drops of tincture of guaiacum. Raw milk will produce a blue coloration if present.

**Analysis of Altered Milk (Maceration Process).**—The total contents of the bottle are transferred to a suitable vessel and thoroughly mixed with a wire whisk. From 10 to 12 grammes are weighed out into a flat-bottomed platinum capsule, which has been previously tared, along with a short glass stirring-rod flattened at the ends.

The weighed portions are neutralized with a decinormal strontia solution, phenolphthalein as indicator. Decinormal soda suits, provided the acidity does not exceed 10 c.c. for 10 grammes milk.

The mixture is evaporated over the water-bath down to a cheesy consistency, and whilst so warm that the fat is still in a liquid condition 20 c.c. ether (specific gravity, 0.720) are poured over the milk solids, which are then carefully stirred for some time with the glass rod.

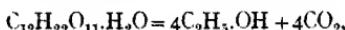
The ethereal solution is now filtered through a tared filter into a tared flask, and the maceration continued with eight successive additions of 10 c.c. of ether—that is, until all the fat has been extracted from the non-fatty solids.

The filter-paper, having its edges cut down, is well washed with boiling ether, and dried at  $100^{\circ}\text{C}$ . The non-fatty solids should now resemble chalk. The capsule is dried overnight, cooled, weighed, and the drying continued until the weight becomes constant.

The correction for the added alkali is in the proportion of 0.0022 gramme per 1 c.c. decinormal soda used, or 0.0042 per 1 c.c. decinormal strontia.

The ethereal solution contained in the tared beakers is evaporated and the weight of fat is ascertained.

When the decomposition has set in to any extent the determination of the amount of alcohol present is the most important item. When a sample measures 6 ounces (170 c.c.), 75 grammes are taken for the estimation of the alcohol by distillation. This portion (75 grammes) is neutralized with soda up to one half of the total acid present. If nearly neutralized, ammonia will pass into the distillate. The first distillate is returned to the flask, with the addition of 0.5 c.c. decinormal soda, and after making up to the original volume, the specific gravity is accurately taken in a bottle at  $15.5^{\circ}\text{C}$ . Now, since lactose is converted into alcohol and carbon dioxide on fermentation, according to the equation—



the percentage by weight of alcohol thus found, multiplied by  $90/46$ , gives the percentage amount of lactose which has been decomposed in the production of the alcohol.

The volatile acid (expressed as acetic acid) is estimated as follows: Ten grammes of the milk are neutralized in a platinum dish to the extent of one half of the total acidity with decinormal soda and a few drops of phenolphthalein added. The contents of the dish are then evaporated to dryness on a water-bath with frequent stirring, treated with 20 c.c. boiling distilled water, and neutralized with decinormal soda. The difference between the number of c.c. of soda originally used and that used for neutralizing the dried substance is equivalent to the amount of volatile acid present. This number multiplied by 0.0255 gives the loss by weight of lactose converted into volatile acid (expressed as acetic acid). The loss incurred by the conversion of proteids into ammonia is small, and may be estimated by diluting 2 grammes of the milk

up to 100 c.c. with distilled water (free from ammonia). The solution is filtered, and 10 c.c. of the clear filtrate, diluted to 50 c.c., is Nesslerized, using a standard solution of ammonium chloride containing 0·01 milligramme ammonia per c.c.

**Analysis of Condensed Milk.**—Weigh out 10 grammes of the thoroughly mixed sample, and dilute to 100 c.c. with water.

**Total Solids.**—Evaporate 20 c.c. of the solution in a flat-bottomed dish in the water-oven, and weigh until constant. This takes about six hours.

**Ash.**—Ignite the residue from the total solids at a dull red heat.

**Proteids.**—Estimate the nitrogen in 10 c.c. by the Kjeldahl process, and calculate the nitrogen into proteids ( $N \times 6\cdot3$ ).

**Fat.**—Estimate the fat in 5 c.c. (in duplicate) by the Adam's coil process.

- **Lactose.**—The most accurate method is to precipitate the proteids with an alkaline solution of copper sulphate, filter, and estimate the reducing sugars in the filtrate by a gravimetric process (Fehling).

A fairly accurate and easy method is to dilute 10 c.c. of the 10 per cent. milk solution to 50 c.c. with water, add 50 c.c. concentrated ammonia, and titrate by Pavy's solution.

### Analysis of Cheese.

**Moisture.**—This is estimated on about 5 grammes of the finely divided sample. Should the sample be very soft, owing to the presence of a large proportion of fat, the amount taken may be mixed with a known quantity of clean sand and dried until the weight becomes constant.

**Fat.**—The dried cheese (mixed with sand) is placed in a Soxhlet tube, and the fat extracted with ether (specific gravity, 0·720) or petroleum ether.

Messrs. Pearmain and Moor have suggested estimating the fat by the Leffmann-Beam process:—Take 2 grammes of the finely divided sample, destroy the proteids by adding 30 c.c. concentrated hydrochloric acid in a shallow dish (milk-dish) on the water-bath. Introduce the mixture into the bottle, rinsing out the dish with 3 c.c. of the hydrochloric acid-fusel-oil mixture, fill up with hot concentrated hydrochloric acid, and rotate as usual. Ten divisions

on the bottle equal 1 per cent. by weight of fat operating upon 15·55 grammes or 15 c.c. milk.

The factor, therefore, becomes for 2 grammes cheese :

$$\frac{15\cdot55}{2} = 7\cdot77.$$

The fat should be further examined for the presence of foreign fats by the processes described.

*Lactose*.—The sugar in cheese may be estimated by macerating with hot water, filtering, and titrating the clear filtrate by Fehling's method.

*Proteids*.—The proteids are usually obtained by difference. They may be estimated in the form of nitrogen by Kjeldahl's process.

#### Analysis of Butter.

**Microscopic Examination.**—Place a small piece of the butter upon a slide, cover it with one drop of olive-oil, and gently press down on this the cover-slip. Examine both under ordinary and polarized light.

Pure butter exhibits characteristic appearances : water will be observed as small globules ; salts and preservatives will be seen under polarized light as bright spots or crystals.

Margarine presents a coarse granular appearance.

**Chemical Examination.**—*Water*.—This is estimated on 5 grammes of the sample placed in a milk-dish and dried till constant in weight in the water-oven.

*Curd*.—Mix the dried butter with about 10 c.c. ether. Filter the ethereal solution through a tared filter; treat the residue repeatedly in this way with ether until all the fat has been extracted, then transfer the whole residue on to the filter, washing it with a little more ether. Dry the paper and contents until the weight becomes constant in the water-oven. The weight obtained represents the amount of curd and salt.

*Ash*.—Ignite the dried filter and contents in a platinum capsule at a dull red heat to prevent volatilization of salts. The residue usually consists almost entirely of salt. The exact amount of salt present may be estimated by titrating its aqueous solution with standard silver nitrate.

*Fat.*—The fat is most accurately estimated by difference, or it may be ascertained by evaporating off the ether from the curd and salt estimation, and weighing the residual fat.

*Preservatives.*—These are estimated as described under milk.

*Colouring Matter.*—Gradually add 4 c.c. of carbon disulphide to 30 c.c. alcohol with gentle agitation till both are thoroughly mixed. Dissolve 5 grammes of the sample in 25 c.c. of this solution in a test-tube. The carbon disulphide will dissolve and carry down with it all the fats, the alcohol, on the other hand, rising to the surface, with any colouring matter that may be present.

After complete separation, which takes only a few minutes, the alcohol solution is removed and evaporated to dryness on a water-bath, and the residue treated with a few drops of strong sulphuric acid. A greenish-blue coloration will indicate the presence of annatto, a pink colour that of a coal-tar dye.

**Detection and Estimation of Foreign Fats in Butter.**—Artificial butters or butter substitutes are manufactured in this country and in the U.S.A., and are called "margarine" and "oleomargarine."

Amongst the most commonly used fats are beef fat, mutton fat, lard, cotton-seed-oil, palm-oil, grease wastes, bone fat, cocoanut-oil, etc.

The clarified fat is compressed, mixed with milk (usually skim milk) and salt, coloured, and occasionally butter added.

In order to examine fats in butter it is necessary to separate them from the other constituents—water, curd, and salt.

**Preparation of the Butter Fat.**—Place about 50 grammes of the butter in a dry beaker and warm it to 50° to 60° C., allowing the water and curd to fall out. Filter the clear fat through a ribbed hot-water jacketed filter, and collect in this way about 30 c.c. in another dry beaker. The fat should be filtered clear, and its temperature must not exceed 70° C.

The specific gravity is next taken at 37.8° C. (100° F.).

The specific gravity of genuine butter fat varies from 0.9105 to 0.9138, and seldom reaches lower than 0.9110. The presence of margarine (lard, etc.) will lower the specific gravity, whilst adulteration with cotton-seed-oil will increase it.

The specific gravity of butter alters on keeping for some time.

**Hehner's Process.**—Butter fat contains less insoluble (in water) acids than other fats.

Four grammes of the prepared butter fat, freed from water and curd, are placed in a tared 8-ounce conical flask, 10 c.c. of saturated alcoholic potash added, and the flask heated gently on a water-bath. Now add a few drops of water, and if any fat remains undissolved, add more alkali and heat again until saponification is complete. Add 50 c.c. water, and evaporate off all the alcohol on the water-bath. Transfer the contents of the flask into a separating-funnel, washing out the flask with a little hot water. Add hot dilute hydrochloric acid (1 : 3) until the solution is acid, and allow to stand for twelve hours in order to effect complete separation of the insoluble fatty acids; tap off the water, leaving the acids adhering to the sides of the separator. Wash the acids in this way several times with water, until the wash-water is free from acid reaction. Dissolve the fatty acids in, and rinse out the separator with, a little ether, tapping the solution into the original tared flask. Evaporate on the water-bath, and continue heating until the weight is constant. The ethereal solution may be evaporated in a tared dish. Animal fats contain about 95·5 per cent. insoluble fatty acids; butter fat contains on an average 87·5 per cent.

Let  $x$  equal per cent. foreign fat and  $y$  equal per cent. insoluble fatty acids found.

$$\text{Then, } x = \frac{100(y - 87\cdot5)}{8} = 12\cdot5(y - 87\cdot5).$$

**Koettstorfer's Method.**—This consists in estimating the molecular weight of the fatty acids—that is to say, the weight of each acid required to neutralize a given weight of potash. This quantity is known as the "Koettstorfer number."

1. *Alcoholic Potash*.—Dissolve 32 grammes potassium hydrate in 1,000 c.c. alcohol. The use of methylated spirit causes discolouration, and the solution must be standardized, in any case, periodically.

2. *Seminormal hydrochloric acid*.

3. *Dilute phenolphthalein solution*.

Weigh out 2 to 3 grammes of the clarified butter fat into an 8-ounce conical flask, add exactly 25 c.c. of the alcoholic potash from a burette, attach the flask to a reflux condenser over a water-

bath, and heat until the fats have been completely saponified. Add a few drops of the phenolphthalein solution, and titrate with the hydrochloric acid. The end-point is sharp, and is indicated by a change in the colour to a yellow tint. A blank titration is

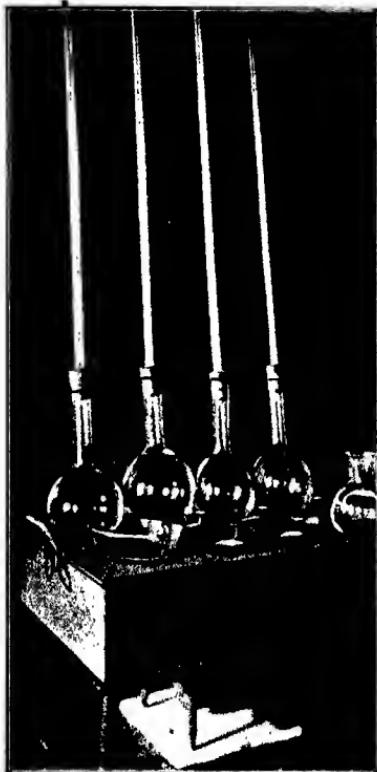


FIG. 24.—WATER-BATH WITH FLASKS PROVIDED WITH A SIMPLE REFLUX CONDENSER ARRANGEMENT (KOEITSTORFER PROCESS).

also carried out upon another 25 c.c. of the alcoholic potash, and the difference between the number of c.c. of acid required in each case noted. This difference represents the amount of potash neutralized by the fatty acids, and when multiplied by 0·028 equals

the weight of potassium hydrate neutralized by the amount of butter fat taken. This weight, again, divided by the amount taken, gives the Koettstorfer number. *E.g.*—

$$\begin{aligned} \text{Fat taken} &= 2 \text{ grammes} \\ 25 \text{ c.c. KOH} &= 22.1 \text{ c.c. HCl.} \\ 25 \text{ c.c. KOH (after treating 2 grammes butter)} &= 6.68 \text{ c.c. HCl,} \\ \therefore 16.42 \text{ c.c. HCl} &= \text{the fatty acids in 2 grammes butter fat.} \\ = 8.21 \text{ c.c. HCl} &= 8.21 \times 0.028 = 230 \text{ milligrammes KOH.} \end{aligned}$$

The Koettstorfer number for pure butter fat varies between 221 and 223 (average, say, 227.25), oleo-margarine being approximately 195.

Let  $n$  equal the Koettstorfer number, then an approximate estimate of the percentage of foreign fat is obtained by the formula

$$(227.25 - n) \times 3.17.$$

**The Reichert-Wollny Process.**—Five grammes of the clarified fat are placed in a 300 c.c. flask (as illustrated), along with 2 c.c. of a 50 per cent. aqueous solution of NaOH, free from carbon dioxide, and 10 c.c. of 96 per cent. alcohol, and connected with a reflux condenser over boiling water for fifteen minutes. The alcohol is then distilled off by disconnecting the flask and leaving it on the bath for about half an hour, when a dry soap will be obtained. 100 c.c. of hot water (distilled) are next added, and the flask and contents boiled for another fifteen minutes until the soap is completely dissolved. The flask is next cooled down to about 60° C., and 40 c.c. normal sulphuric acid added, along with a small piece of platinum coil or coiled wire. It is much more preferable in all cases to use platinum scrap to prevent bumping than pumice or pipe-clay. The flask is then connected with the Liebig condenser and warmed, gently at first. The temperature having been gradually raised, it is so adjusted that 110 c.c. distil over in the course of half an hour. The distillate is thoroughly mixed, and 100 c.c. are filtered off into another vessel, 1 c.c. phenolphthalein solution (1 gramme in 1,000 c.c.) added, and titrated with decinormal sodium or barium hydrate. The number of c.c.'s of alkali used is multiplied by 1.1, a blank distillation being also carried out under similar conditions, the number of c.c.'s of alkali used in which should not exceed 0.33.

*Formula.*—Calculation of true fat in butter where  $x$  is the Reichert value and 28.78 the Reichert value of genuine butter fat—

$$F = \frac{100(x - c)}{28.78 - c}.$$

For convenience, the value of  $c$  (the foreign fat) may be taken as = 3.

**Zeiss Butyro-Refractometer.**—This instrument is operated in either diffused sunlight or in artificial light. A constant stream

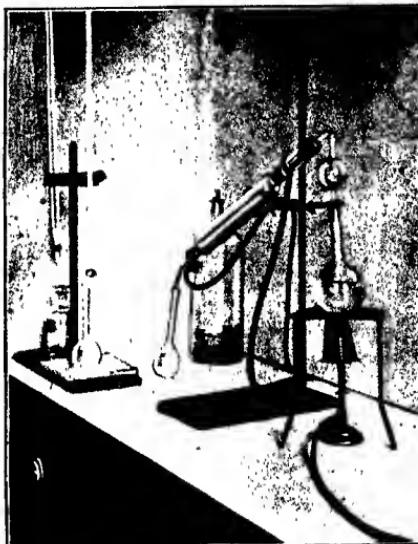


FIG. 25.—REICHERT-MEISSL-WOLLNY APPARATUS.

of water of even temperature is passed through the nipple  $D$ . Half turn the pin  $F$ , and open out the segment  $B$ , which moves upon the pinion. This done, the prisms are now most carefully cleansed from all grease and dust by lightly rubbing with a soft rag containing a little ether. Place a drop or so of the clarified fat upon the movable prism in the jacket  $B$  in such a way as to secure a thin but even layer. Fold back and compress the prism  $B$  against the prism  $A$ , and lock it by half turning the screw  $F$ .

On observing through the telescope, tilt the mirror  $J$  till the

critical line, in the centre of the field, is rendered sharp, after ascertaining that the faces of the prisms are uniformly smeared with the fat, and then focus the eyepiece of the telescope as may be required.

It will be noticed that the critical line becomes more distinct

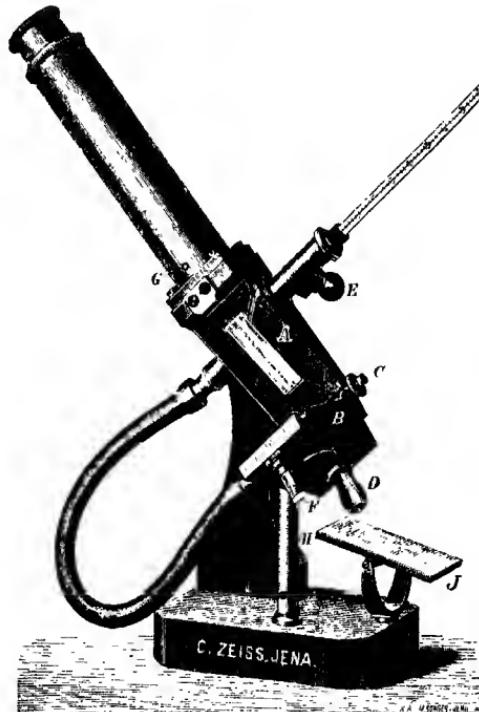


FIG. 26.—ZEISS BUTYRO-REFRACTOMETER.

after a period of some seconds, after which it becomes constant. The colour, if any, is then noted, together with the position of the critical line on the centesimal scale, the temperature at the moment also being read.

Further directions, together with a standard fluid, are supplied with each instrument, as it is necessary to standardize it from time to time.

## Table of Results (Wollny).

Fat.	Temperature.	Scale Divisions.	Refractive Index D.
Genuine butter ..	25° C.	49·5 to 54·0	1·4500 to 1·4620
Margarine ..	25° C.	58·6 to 66·4	1·4650 to 1·4700

From this it will be seen that all readings higher than 54·0 must be considered as showing adulteration in the case of butter.

An average limit may be therefore taken as at 52·5 (25° C.) scale divisions for the critical line.

The scale divisions are converted into refractive indices according to the table :

Reading.	Refractive Index D.	Reading.	Refractive Index D.	Reading.	Refractive Index D.
00·0	1·4220	52·5	1·4609	66·5	1·4704
10·0	1·4300	53·0	1·4613	67·0	1·4707
20·0	1·4377	53·5	1·4616	67·5	1·4710
30·0	1·4452	54·0	1·4619	68·0	1·4713
40·0	1·4524	54·5	1·4623	68·5	1·4717
40·5	1·4527	55·0	1·4626	69·0	1·4720
41·0	1·4531	55·5	1·4629	69·5	1·4723
41·5	1·4534	56·0	1·4633	70·0	1·4726
42·0	1·4538	56·5	1·4636	70·5	1·4729
42·5	1·4541	57·0	1·4639	71·0	1·4732
43·0	1·4545	57·5	1·4642	71·5	1·4735
43·5	1·4548	58·0	1·4646	72·0	1·4738
44·0	1·4552	58·5	1·4649	72·5	1·4741
44·5	1·4555	59·0	1·4652	73·0	1·4744
45·0	1·4558	59·5	1·4656	73·5	1·4747
45·5	1·4562	60·0	1·4659	74·0	1·4750
46·0	1·4565	60·5	1·4662	74·5	1·4753
46·5	1·4569	61·0	1·4665	75·0	1·4756
47·0	1·4572	61·5	1·4669	75·5	1·4759
47·5	1·4576	62·0	1·4672	76·0	1·4762
48·0	1·4579	62·5	1·4675	76·5	1·4765
48·5	1·4583	63·0	1·4678	77·0	1·4768
49·0	1·4586	63·5	1·4681	77·5	1·4771
49·5	1·4590	64·0	1·4685	78·0	1·4774
50·0	1·4593	64·5	1·4688	78·5	1·4777
50·5	1·4596	65·0	1·4691	79·0	1·4780
51·0	1·4600	65·5	1·4694	80·0	1·4783
51·5	1·4603	65·5	1·4697	90·0	1·4840
52·0	1·4606	66·0	1·4700	100·0	1·4895

## CHAPTER X

### OILS AND FATS

#### Composition and Properties.

FATS and oils are glycerides or glycerine salts of the fatty acids. The commoner examples are olein, palmitin, and stearin, being the triglycerides of oleic, palmitic, and stearic acids. They are insoluble in water, more or less soluble in pure alcohol, and easily soluble in ether, chloroform, amyl alcohol and carbon bisulphide. They are easily decomposed by alkalies yielding soaps and free glycerine.

The triglycerides, or neutral salts, are found in animal and vegetable products, the mono- and di-glycerides being synthetical (artificial) formations.

The following is a list of edible fats and oils (Lewkowitsch) :

#### Acids of the Acetic Series— $C_nH_{2n}O_2$ .

Name.	Formula.	Melting-Point.	Boiling-Point.	Occurrence.
Butyric	$C_4H_8O_2$	$-2^{\circ}-+2^{\circ}$	$162\cdot3^{\circ}$	Butter, cocoa butter.
Caproic	$C_6H_{12}O_2$	—	$200^{\circ}$	Butter, cocoanut-oil.
Caprylic	$C_8H_{16}O_2$	$16\cdot5^{\circ}$	$236^{\circ}$	Butter, cocoanut-oil.
Capric	$C_{10}H_{20}O_2$	$31\cdot3^{\circ}$	$268-270^{\circ}$	Butter, cocoanut-oil.
Lauric	$C_{12}H_{24}O_2$	$43\cdot6^{\circ}$	$176^{\circ}$	Cocoanut-oil, cocoa butter.
Myristic	$C_{14}H_{26}O_2$	$53\cdot8^{\circ}$	$196\cdot5^{\circ}$	Cocoanut-oil, sesame-oil.
Palmitic	$C_{16}H_{32}O_2$	$62^{\circ}$	$215^{\circ}$	Almost all fats and oils.
Stearic	$C_{18}H_{36}O_2$	$71^{\circ}-71\cdot5^{\circ}$	$232\cdot5^{\circ}$	Fats, and nearly all oils, except corn and olive.
Arachidic	$C_{20}H_{40}O_2$	$77^{\circ}$	—	Peanut, olive (trace), rap (trace).
Behenic	$C_{22}H_{44}O_2$	$83^{\circ}-84^{\circ}$	—	Rape, mustard.
Lignoceric	$C_{24}H_{48}O_2$	$81^{\circ}$	—	Peanut.

Acids of the Oleic Series— $C_nH_{2n-2}O_2$ .

Name.	Formula.	Melting-Point.	Boiling-Point.	Occurrence.
Hypogaeic	$C_{16}H_{30}O_2$	33°	236°	Peanut.
Oleic	$C_{18}H_{32}O_2$	14°	232.5°	Almost all fats and oils.
Iso-oleic	$C_{18}H_{34}O_2$	44°-45°	—	—
Rapic	$C_{18}H_{34}O_2$	—	—	Rape and mustard.
Erucic	$C_{22}H_{42}O_2$	33°-34°	264°	Rape and mustard.

Acids of the Linoleic Series— $C_nH_{2n-4}O_2$ .

Name.	Formula.	Melting-Point.	Boiling-Point.	Occurrence.
Linoleic	$C_{18}H_{32}O_2$	Under 18°	—	Olive, cotton-seed, peanut, sesame, cocoa butter, poppy-seed, sunflower.

Animal fats and oils are usually extracted by means of heat, dry or steam, either with or without pressure ; whilst vegetable products are either treated with solvents or pressure alone.

**Moisture.**—Water is extracted, if present, by allowing it to separate out on standing the melted fats in a warm place for some time, and decanting off the clear fat or oil. The amount of moisture present may be determined by drying a weighed quantity of the fat in a shallow dish in the steam-oven.

**Weighing Fats.**—The fat may be pipetted off while molten, or it may be cooled over ice, and weighed upon a thick tared filter-paper placed upon a tared dish ; both paper and fat may be transferred together to a flask or other apparatus.

**Specific Gravity.**—The specific gravity of liquid oils is usually taken at 15.5° C. by means of a hydrometer, Westphal balance, Sprenge tube, or gravity-bottle. Solid fats may be taken at temperatures exceeding their melting-points, or at 100° C., by means of the gravity-bottle or balance.

The coefficient of expansion with increasing temperature for all oils and fats is the same, the mean absolute coefficient of ex-

pansion for  $1^{\circ}$  being approximately 0·0008, and the apparent coefficient of expansion almost 0·00077.

**Viscosity.**—The viscosity or fluidity of edible fats and oils, although seldom required, may be ascertained by means of any of the well-known types of viscosimeters.

**Melting-Point.**—A capillary tube is drawn out from a piece of small glass tubing, and its open end is immersed in a beaker of the

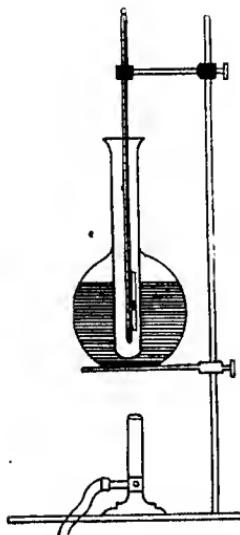


FIG. 27.—APPARATUS FOR DETERMINING THE MELTING-POINT (CAPILLARY METHOD).

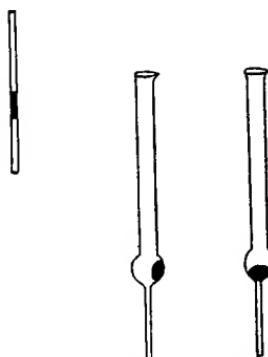


FIG. 28.—IMPROVED CAPILLARY TUBE FOR DETERMINING THE MELTING-POINT OF FAT.

molten fat, when the fat will rise up into the capillary bore. The tube is now allowed to set for about twelve hours, and is thereafte attached by a rubber band to a thermometer graduated in tenths the fat particle being close to the bulb of the thermometer. Both are then sunk in a test-tube containing water, which is also held in a boiling-flask partially filled with water, as shown in the accompanying diagram. Heat is then applied gradually, so that the thermometer rises not more than 0·5° per minute, until the fat fuses

the exact temperature being that of the melting-point of the fat. Several observations are made on the same sample, the average result being accepted.

**Solidifying - Point.**—The point of solidification of the fat is observed after that of its melting-point.

**Refraction.**—The comparison of the refractive power of fats and oils forms a most important part of their analysis. The degree of deflection caused in a ray of light passing from one transparent medium into another is known as the refractive index of that medium, and is the quotient of the sine of the angle of the incident divided by the sine of the angle of the refracted ray.

A further description of the refractometer and its use is described under Butter.

**Polarization.**—Oils are either dextro- or laevo-rotatory, whilst pure glycerides are usually optically inactive.

Before observation all oils must be rendered clear and limpid by filtration or other suitable means.

**Microscopical Examination.**—This is of use more particularly in the case of solid fats. Crystals—*e.g.*, beef or lard—may be obtained by slow crystallization at ordinary temperatures from ether, which may be washed with alcohol, or alcohol and ether, and recrystallized from ether.

**Polarized Light.**—Fat crystals, obtained as above described, or by merely allowing the melted fat to cool slowly, often present characteristic appearances under polarized light, with or without selenite plates, sufficiently marked as to allow of the detection of various adulterants.

**Spectroscopic Examination.**—By this method the presence of chlorophyll and its products may be demonstrated, especially if the sample is compared with one or more of known purity.

**Turbidity Temperature (Valenta).**—The fat is dissolved in glacial acetic acid with the aid of heat, and then allowed to cool slowly, the temperature at which turbidity ensues being noted. Three c.c. of the fat should be dissolved in 3 c.c. of glacial acetic acid. The following are a few representative turbidity numbers: Butter fat, from 40° to 70° C., averaging 52° to 65° C.; oleomargarine, 95° to 106° C.; rape-oil, 101° C.; linseed-oil, 53° to 57° C.; sesame-oil, 77° C.; peameal-oil, 61° to 88° C.; olive-oil, 89° C.; and lard-oil, 96° C.

**Volatile Fatty Acids (Reichert-Meissl Process).**

Five grammes of the fat are placed in a dry Erlenmeyer flask, 300 c.c. capacity, 10 c.c. of 95 per cent. alcohol added, and 2 c.c. sodic hydrate solution (100 grammes NaOH in 100 c.c. H<sub>2</sub>O). The flask and contents are then heated on a water-bath, under a reflux condenser, with occasional agitation, till the saponification is completed. This will be evident by the absence of any fat globules. The contents of the flask are then thoroughly dried by prolonged heating upon the water-bath, and, if necessary, whirling in the air to expel all traces of alcohol and moisture; 135 c.c. of distilled water are then added, in which the soap is dissolved by gentle warming. The flask is cooled, and 5 c.c. dilute sulphuric acid (200 c.c. in 1,000 c.c. water) are added, and the emulsion again warmed, to effect thorough admixture and solution, without access to the air. A piece of platinum foil may now be added to prevent "bumping," and the flask then connected with a condenser and receiver, as described under Butter.

**Estimation of Soluble and Insoluble Fatty Acids—Soluble.**

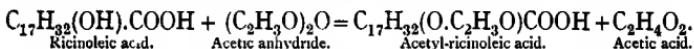
—Five grammes of the fat are weighed out as in the Reichert-Meissl process, and 50 c.c. alcoholic potash solution (40 grammes KHO in 1,000 c.c. of 95 per cent. redistilled alcohol) are added from a burette, and the contents of the flask completely saponified as described in that process, after which the alcohol is expelled by further heating. Several blank experiments are also simultaneously started, and are titrated, after evaporation, with  $\frac{N}{2}$  hydrochloric acid. One c.c. in excess of the number which is required to neutralize the blanks is then added to the fatty acids, and the flask again heated under a reflux condenser until all the fatty acids have separated out. The flask having been thoroughly cooled in running water, and the fatty acids thus solidified, the clear solution is carefully decanted off through a tared filter into a litre flask; 200 c.c. to 300 c.c. warm water are then added to the residual fatty acids in the flask, which are again melted on the water-bath and again cooled, and the process of washing by decantation repeated three times. The combined washings are made up to 1,000 c.c. with water, thoroughly mixed, and two portions of 100 c.c. each are titrated with  $\frac{N}{10}$  NaOH, using phenolphthalein as the indicator. The readings, multiplied by

to represent the amount of  $\frac{N}{10}$  alkali required to neutralize the acidity of the soluble fatty acids, together with the excess (1 c.c.) of  $\frac{N}{2}$  acid used. As this 1 c.c.  $\frac{N}{2}$  acid is equivalent to 5 c.c.  $\frac{N}{10}$  alkali, 5 c.c. must be deducted from the number of c.c.'s alkali used in effecting neutralization of the fatty acids. This corrected number, after being multiplied by 0·0088, will represent the weight of soluble fatty acids in the 5 grammes originally taken in terms of butyric acid.

**Insoluble.**—Transfer the fatty acids remaining in the flask, in a cake, to a weighed glass evaporating-basin, with the aid of alcohol. Also wash the residue left in the tared filter into the basin with alcohol, and evaporate off the alcohol upon the water-bath, drying the contents of the dish in the water-oven for two hours; cool, and weigh. Again heat the acids for half an hour, cool, and again weigh.

**Free Fatty Acids.**—Weigh 20 grammes of the fat or oil into a 150-c.c. Erlenmeyer flask, and add 50 c.c. of 95 per cent. alcohol (previously neutralized with dilute NaHO, using phenolphthalein as the indicator). Warm to 60°, and titrate with  $\frac{N}{10}$  sodic hydrate, using phenolphthalein. Each c.c.  $\frac{N}{10}$  alkali is equivalent to 0·0282 grammie oleic acid, or as the "acid-number" of c.c. of  $\frac{N}{10}$  alkali required to neutralize the free fatty acids in 1 grammie of the fat or oil under examination.

**The Acetyl Value.**—Fats or oils are "acetylated" when heated with acetic anhydride—*i.e.*, the hydrogen atom of the alcoholic hydroxyl group is exchanged for the acetic acid radicle:—



The acetyl value is therefore the number of milligrammes of potassium hydrate required to neutralize the acetic acid formed by the saponification of 1 grammie of the acetylated fat.

Ten grammes of the fat or oil are boiled with 10 c.c. acetic anhydride for two hours under a reflux condenser, and then in an open beaker with 500 c.c. water for half an hour. The solution is allowed to separate out, the lower liquid is siphoned off, and the oily layer repeatedly boiled, and thus washed, with fresh water till free from acid (litmus reaction). The acetylated fat is then removed and

dried in the water-oven, and from 2 to 4 grammes weighed into a flask and saponified with alcoholic potash, as in the Koettstorfer process (saponification number). The alcohol is evaporated off, the soap dissolved in water, and the free acetic acid either distilled or filtered off, and titrated as follows :—

1. The acetic acid may be distilled off, after having first acidified the soap with 1 : 10 sulphuric acid, receiving from 600 to 700 c.c. distillate by occasionally adding water to the distillation flask. The distillate is filtered as distilled through a loose cotton-wool plug to free it from any distilled insoluble acids, and is titrated with  $\frac{N}{10}$  NaOH, using phenolphthalein. The number of c.c.'s of alkali used, multiplied by 5·61, and the product divided by the number of grammes of acetylated fats taken, represents the acetyl value.

2. The filtration process is more rapid, and as accurate as the distillation process. The quantity of alcoholic potash used in originally saponifying the fat should be exactly neutralized by adding standard acid. This may easily be ascertained by blank experiments. The resulting mixture is then warmed, when the fatty acid will separate out upon the top of the liquid, and may be filtered off, washed free from acid with boiling water, and the filtrate titrated as above with  $\frac{N}{10}$  NaOH, the number of c.c.'s of which are calculated as described into the acetyl value.

**Saponification Equivalent (Koettstorfer Process).**—The saponification number or equivalent is the number of milligrammes of potassium hydrate required to completely saponify 1 gramme of the fat or oil.

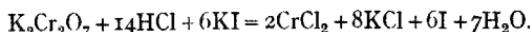
From 1 to 2 grammes of the fat are transferred to an Erlenmeyer flask, and 25 c.c. alcoholic potash (40 grammes KHO in 1,000 c.c. of 97 per cent. alcohol) are rapidly added from a burette.

Duplicate experiments are simultaneously carried out, along with blanks. The saponification is effected upon the water-bath, and the excess of alkali is titrated, after cooling, with  $\frac{N}{2}$  HCl, using phenolphthalein. The number of c.c.'s of  $\frac{N}{2}$  acid required to neutralize the blanks is subtracted from that taken in the case of the fats, and the difference multiplied by 28·06, and divided by the number of grammes originally taken, represents the saponification number or equivalent.

**Iodine Absorption—Hubl's Method.**—Prepare the following solutions :—

1. Iodine solution : By dissolving 26 grammes iodine in 500 c.c. of 95 per cent. alcohol, and, separately, 30 grammes mercuric chloride in a like quantity of the alcohol. Filter the mercuric solution, if necessary ; mix it thoroughly with the iodine, and allow to stand at least twelve hours previous to use.
2. Decinormal thiosulphate solution : By dissolving 24·6 grammes pure "hypo," and making up to 1,000 c.c. in water.
3. Potassium iodide solution : By dissolving 150 grammes in water, and diluting to 1,000 c.c.
4. Potassium dichromate solution : By dissolving 3·874 grammes of the pure salt in water, and making up to 1,000 c.c.
5. Starch paste : By dissolving 1 gramme starch into 200 c.c. water, and cooling the liquid.

Standardize the thiosulphate solution by running it into a flask containing 20 c.c. of the dichromate solution, 10 c.c. potassium iodide, and 5 c.c. concentrated hydrochloric acid, until only a faint straw-coloured liquid is obtained ; then add a little of the starch, and exactly decolourize the blue solution thus arrived at according to the equation :



The equivalent of 1 gramme of iodine in terms of the thiosulphate is obtained by multiplying the number of c.c.'s of the thiosulphate solution required for titration by 5. Thus :—If 16·6 c.c. thiosulphate were used for 20 c.c. dichromate, then 1 gramme of iodine is equivalent to  $16\cdot6 \times 5 = 80\cdot0$  c.c. thiosulphate solution, or 1 c.c. of that solution will equal  $\frac{1}{80\cdot0} = 0\cdot01123$  gramme iodine.

One c.c. strictly  $\frac{1}{16}$  thiosulphate solution is theoretically equivalent to 0·0127 gramme iodine.

*Process.*—Take 1 gramme of a solid fat, or 0·2 to 0·4 gramme of an oil, in a well-stoppered flask or bottle of 300 c.c. capacity, and dissolve it in 10 c.c. chloroform. Add 30 c.c. of the iodine from a burette, and allow to stand in the dark, with occasional agitation, for three hours. Then add 20 c.c. potassic iodide and 100 c.c. distilled water, and titrate, as already described for standardization,

with the thiosulphate solution, finishing off with starch indicator. Towards the end of the titration the liquid must be thoroughly mixed by shaking vigorously, in order to dissolve any particles of iodine, and the blue colour should not reappear after an interval of five minutes. The experiment should be carried out in duplicate, and along with two blanks.

*Example.*—In one experiment 30 c.c. of the iodine required 54·2 c.c. of the thiosulphate solution, and in another 54·8 c.c. The average or mean is 54·5. Again, 16·2 c.c. of thiosulphate were required for the excess of iodine unused (or unabsorbed) by 1 gramme of the fat. Then the thiosulphate equivalent to the iodine actually absorbed by the fat would be  $54\cdot5 - 16\cdot2 = 38\cdot3$  c.c., and the per cent. of iodine thus absorbed by the fat would be  $38\cdot3 \times 0\cdot01123 \times 100 = 47\cdot10$ .

**Hanus' Method.**—As the Hubl method requires considerable time, and is inconvenient in that the solution of iodine is unstable and loses in its strength, this (Hanus') method, and the following of Wij's, are found more satisfactory in use.

*Solutions.*—Iodine solution : Dissolve 13·2 grammes iodine in 1 litre glacial acetic acid (99 per cent.), and add, when cold, 3 c.c. bromine, or at least sufficient to approximately double the halogen content, leaving a slight excess of iodine.

Prepare the  $\frac{1}{10}$  thiosulphate of soda, potassium iodide, and starch solutions, as in the Hubl method.

*Process.*—Proceed as described in the Hubl process, using 30 c.c. of the iodine (Hanus); mix well before adding water previous to titrating, and use only 10 c.c. potassium iodide. The absorption requires thirty minutes, as compared with three hours, as in the case of the Hubl. Due precaution must be taken that the iodine solution is always measured out at the same temperature, both for assay and blank experiments, as the concentrated acetic acid has a very high coefficient of expansion; otherwise a large discrepancy may be incurred.

**Wij's Method**—*Solution.*—Dissolve 13·2 grammes iodine in 1,000 c.c. glacial acetic acid, and pass through a very large portion of this solution a current of pure dry chlorine until the liquid is almost decolourized. Then add the remainder of the iodine solution, until the red iodine colour is again obtained, thus practically

doubling the halogen content, with a slight excess of iodine. Prepare the other solutions as in the Hubl method.

*Process.*—Proceed as in the Hanus method, taking for non-drying oils and fats fifteen minutes, for semi-drying oils thirty minutes, and for drying oils one hour.

**Maumené Test.**—Place a 150-c.c. beaker within a large beaker, packing the intervening space with a non-conducting medium, such as cotton-waste or wool. Weigh into the inner beaker 50 grammes of the oil, and note its temperature after replacing it in position in the nest. Fill a burette with concentrated sulphuric acid possessing the same temperature as the oil, and run 10 c.c. of it into the oil

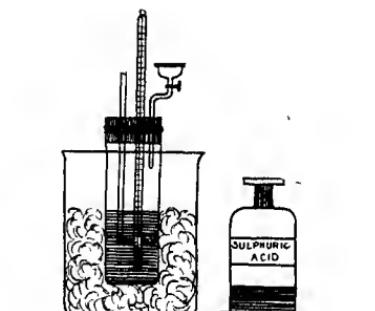


FIG. 29.—APPARATUS FOR DETERMINING THE RISE OF TEMPERATURE : MAUMENÉ TEST.

with constant stirring, noting the consequent rise in temperature of the mixture, of which the maximum point is recorded.

The Maumené number is the difference in degrees C. between the initial and maximum temperatures.

Should excessive frothing ensue, as is the case with many oils—e.g., cotton-seed—the acid must be diluted. The result obtained by dividing the rise of temperature in the case of the oil by the rise of temperature in the case of water, with the same strength of acid, and multiplying the quotient by 100, will give what is termed as “the specific temperature reaction.”

**Bromination Test.**—This test consists in ascertaining the rise in temperature caused by the reaction and absorption of bromine by fats and oils. Although the actual amount of absorption is not

obtained, yet the results obtainable bear a close relationship with those of the iodine numbers : and as the test is more convenient than that of the iodine absorption already described, it may in some instances be carried out in preference to the latter, and the results obtained calculated, by factors, into iodine values.

The Gill and Hatch apparatus consists of a long, narrow, flat-bottomed tube, supported by a cork in a beaker, which is placed in a cotton-wool or waste nest within a second and larger beaker.

Five grammes of the substance are dissolved in 25 c.c. chloroform or carbon tetrachloride, and of this solution 5 c.c. are pipetted into

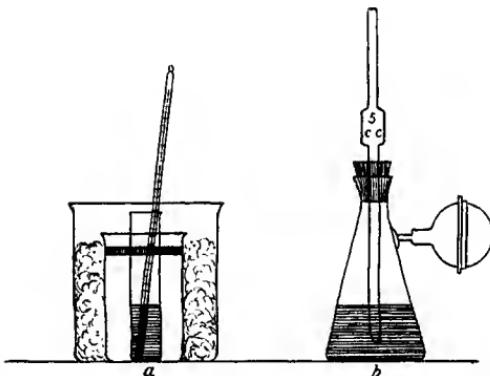


FIG. 30.—*a*, GILL AND HATCH'S CALORIMETER FOR THE BROMINATION TEST FOR OILS; *b*, WILEY'S PIPETTE FOR MEASURING BROMINE IN CHLOROFORM.

the inner tube in such a way that none of the liquid is spilt upon its sides. The temperature is next observed with a thermometer graduated in tenths of a degree. A bromine solution, freshly prepared by adding 1 volume to 4 volumes of chloroform or carbon tetrachloride from a burette, is poured into a Wiley's bromine pipette, which consists of a side-necked filter-flask fitted with a rubber cork, through which passes a 5-c.c. pipette and exhaust-bulb fitted on the side neck.<sup>4</sup> By compressing the bulb, 5 c.c. of the bromine reagent can be with convenience pipetted, with stirring, into the dissolved substance already placed in the calorimeter. The maximum degree of rise in temperature is noted, as in the

Maumené test, the difference between the initial and maximum temperatures being stated as that of "the heat of bromination number." The experiment should be repeated until concordant results are obtained.

For convenience of comparison, a standard bromination liquid may be prepared by, for example, dissolving 7·5 grammes sublimed camphor in 25 c.c. carbon tetrachloride, and pipetting of this solution the usual 5 c.c. for bromination. In this way an average rise in temperature of about 4·2° C. should be obtained for the standard, the specific temperature reaction for each oil being obtained by dividing the heat of bromination by this number.

From experiment it has been ascertained that, by dividing the iodine number of various oils by the specific bromination temperature reaction, the factor required for converting the latter value into the iodine number was 17·18.

**Elaidin Test.**—Liquid olein is converted into solid elaidin by the action of nitrous oxide, which is a crystalline compound isomeric with olein, other common glycerides remaining liquid. The test involves determining the consistency of the resulting mass after such a reaction.

**Process.**—Place 5 grammes of the fat or oil in a beaker, add 7 grammes nitric acid (specific gravity, 1·34), and about 0·5 grammes copper wire or turnings. Warm the beaker and contents to 15° C., and thoroughly mix the nitrous oxide bubbles with the liquid by stirring. Set aside for two hours, noting the time taken for solidification.

Lard-oil, pure olive-oil, almond or peanut-oils will turn into a solid white mass. Almost all the seed oils, particularly cotton-seed and mustard-seed, will turn into a pasty consistency.

#### Free Fatty Acids—Solidifying-Point or Titer Test.

Set free the fatty acids—*vide Free Fatty Acids*—from water by filtering, and allow to dry somewhat by standing in a porcelain dish for twelve hours in a desiccator. Melt the mass, and pour into a warm test-tube 3·5 centimetres in diameter, until it is half full. Insert the tube, by means of a wide-bored rubber cork, into the neck of a flask, and introduce a warm delicate thermometer into

the mass, moving it gently upwards and downwards in order to mix it thoroughly. The temperature will fall at first, and then slightly rise, remaining so for a very short period before again falling. The stationary point is taken as "the solidifying-point or titer."

**Unsaponifiable Matter.**—Place 10 grammes of the fat or oil in a 250-c.c. flask, and saponify with a mixture of 25 c.c. alcoholic potassium hydrate and 25 c.c. alcohol under a reflux condenser. Dissolve the soap in about 40 c.c. warm water. Cool, and transfer the whole to a separating funnel, washing out the flask first with 50 per cent. alcohol, and then petroleum ether. Shake up the mixture very gently with a rotary movement, in order to avoid forming an emulsion. An emulsion may be broken up by adding a little water (cold), or a little ether or alcohol. Allow the ethereal solution to separate out completely, and run off the soap solution. Wash the ethereal solution several times with 50 per cent. alcohol, adding the washings to the soap solution in another separator, and finally wash the combined soap solution and washings twice with petroleum, and then add the ethereal washings to the main ethereal solution. Run the ethereal solution and washings into a tared flask, evaporate off all traces of ether, and weigh the residual unsaponifiable matter. In the presence of mineral oils, as certain of these are volatile, care must be taken to prevent any loss of such by undue heating.

### Cholesterol and Phytosterol.

Both are monatomic alcohols, combining with fatty acids to form esters.

Cholesterol ( $C_{26}H_{44}O$ ) forms white monoclinic laminae of melting-



FIG. 31.—CHOLESTEROL CRYSTALS UNDER THE MICROSCOPE (AFTER BÖMER.)

point  $145^{\circ}$  and specific gravity 1.067. It is neutral, tasteless, and odourless, insoluble in water, soluble in ether and hot alcohol. Cholesterol occurs in animal fats and oils.

Phytosterol ( $C_{28}H_{44}O.H_2O$ ) crystallizes in thin plates from ether or chloroform, and in needle-shaped tufts from alcohol, with melting-point about  $132^{\circ}\text{ C}$ . It is somewhat akin to cholesterol in its properties.

**Estimation of Cholesterol and Phytosterol.**—Boil 50 grammes of the oil for five minutes in a flask connected with a reflux condenser with two separate portions of 75 c.c. of 95 per cent,

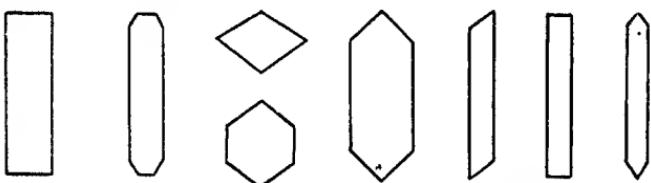


FIG. 32.—PHYTOSTEROL CRYSTALS (AFTER BOMER).

alcohol, removing the alcohol by a separating-funnel. Concentrate the combined alcoholic liquors in a flask to one-fourth this volume, transfer to a dish, and evaporate to dryness. Extract the dry

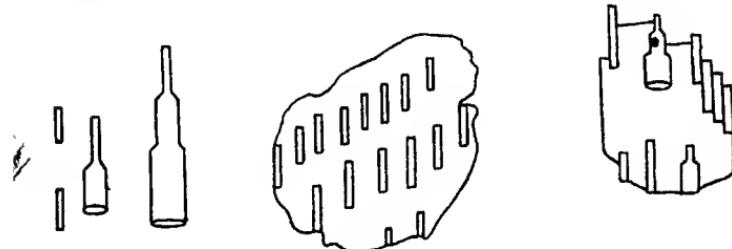


FIG. 33.—MIXED CHOLESTEROL AND PHYTOSTEROL CRYSTALS (AFTER BOMER).

residue with ether, and again evaporate to dryness; repeat the extraction and evaporation, dissolve in hot 95 per cent. alcohol, crystallize out the cholesterol or phytosterol, and weigh. The crystals should afterwards be examined microscopically.

#### Detection and Identification of Oils and Fats.

Cases may arise where it is important to know, for example, whether the oil or fat is of an animal or vegetable nature, so that,

besides having recourse to the processes already described, the analyst must apply various discriminating tests, a few of which will presently be given. Of these, the colorations produced by the oxidizing action of nitric acid (concentrated), by silver nitrate and phosphomolybdic acid, are very useful in assessing animal or vegetable products, especially when taken into full consideration along with the data obtained by, for example, the iodine value, saponification number, and Reichert-Meissl figure ; besides which further information is afforded, often in more obscure cases, by the specific gravity, melting-point, refractive index, and other physical properties.

**Cotton-seed-Oil**—*Becchi's Silver Nitrate Test (Hehner's Modification).*—Two grammes of silver nitrate are dissolved in 200 c.c. 95 per cent. alcohol, free from reducing agents, 40 c.c. ether added, and the reagent very slightly acidified with nitric acid. Ten c.c. of the melted oil or fat are mixed with 5 c.c. of the solution in a test-tube or porcelain dish, and heated on the water-bath for fifteen minutes.

The presence of cotton-seed-oil is demonstrated by a greater or less reduction of the nitrate. Various rancid oils free from cotton-seed, butter fat, and lard derived from animals fed on cotton-cake and fats which have been overheated, give slight reductions with this reagent.

*Becchi's Test (Millau's Modification).*—When doubt arises in the above test, this method of testing the fatty acids is preferable. Saponify 20 grammes of the oil or fat with 30 c.c. alcoholic potash in the usual manner, distilling off the alcohol, and dissolving the soap thus formed in 250 c.c. of hot water. Slightly acidify with 10 per cent. sulphuric acid, and wash the fatty acids, by decantation with water, three times. Apply the Becchi solution as above described.

*Halphen's Test.*—Dissolve equal parts of amyl alcohol and carbon bisulphide (containing 1 per cent. sulphur), and mix it with an equal volume—say 3 to 6 c.c.—of the oil or melted fat in a test-tube, loosely plugged with cotton-wool, and heat in a boiling saturated solution of brine for fifteen minutes.

A deep red or orange colour will be produced by cotton-seed-oil, even as little as 2 per cent. being readily detected in this way, although prolonged boiling affects the results.

Cotton-seed-oil which has been heated to 250° C. does not produce a colour with this reagent.

**Arachis (Peanut) Oil**—*Rénard Test.*—Saponify 5 grammes of the sample with 50 c.c. alcoholic potash (40 grammes per litre 95 per cent. alcohol), neutralize with acetic acid (phenolphthalein indicator), and transfer to a  $\frac{1}{2}$ -litre flask, containing 60 c.c. 10 per cent. lead acetate, with 200 c.c. water. Boil for about a minute, and then cool suddenly; carefully decant off the solution from the soap, and wash by decantation with cold water and then with alcohol. Add 20 c.c. ether, close the flask, and dissolve the soap, with agitation, heating it on the water-bath under a reflux condenser for five minutes. Cool down to 15° to 17° C., and allow to stand for twelve hours. Filter, wash with ether, and again transfer to the same flask with boiling water which has been acidulated slightly with hydrochloric acid, making the whole up to 300 to 400 c.c. with the water.

Allow the fatty acids to separate out, filter, wash with water, and then with 25 c.c. hot 95 per cent. alcohol. As the solution cools, the crystals of arachidic acid will separate out. Filter, and again wash with about 20 c.c. alcohol into another flask; shake thoroughly, again filter, and wash with 70 per cent. alcohol. Dissolve the precipitate in hot absolute alcohol, evaporate to dryness in a tared glass basin, dry, and weigh. Add to the weight thus obtained 0.0025 gramme for each 10 c.c. of 95 per cent. alcohol used in washing and crystallizing, if done at 15° C., and 0.0045 gramme for each 10 c.c. if at 20° C.

The weight of arachidic acid multiplied by 20 indicates, approximately, the amount of peanut-oil present.

The melting-point of the arachidic acid crystals should average 71° to 72° C., and they should also be identified microscopically.

**Sesame - Oil**—*Badouin's Test.*—Dissolve 0.1 gramme cane-sugar in 10 c.c. hydrochloric acid (specific gravity, 1.20) in a test-tube, add 20 grammes of the oil, and shake vigorously for one minute. Allow to stand. The presence of 1 per cent. or more of sesame-oil will be indicated by a red colour in the aqueous solution.

In doubtful cases the test is best carried out upon the fatty acids.

**The Sulphur Chloride Reaction.**—Various oils of vegetable origin

### Table of Constants of Fats and Oils.

Fat or Oil.	Specific Gravity, 15° C.	Melting- Point.	Solidifying- Point.	Reichert- Méissl Number.	Saponifica- tion Number.	Iodine Number.	Insoluble Fatty Acids.	Maurame- Number.	Specific Tempera- ture- Reaction.	Acid Value.	Acetyl Value.
Corn-oil	0.9213-0.9255	—	—	4-5	188-193	113-125	93-96	81-86	6-38	1-3-2-9	—
Cotton-seed-oil	0.9220-0.925	3-4	—	0.95	193-195	108-110	95-96	75-90	1.70	0.0	7.5-18.0
Cotton-seed stearin	0.918-0.923	29-32	16-22	—	195	90-103	96	48	—	—	—
Coconut-oil	0.911	21-24	22-14	70-84	246-260	80-95	185-907	—	—	5-50	1.0-12.2
Cocoa butter	0.9350-0.926	28-33	23-21.5	0.2-0.8	193	32-41	94.6	—	—	1.1-1.8	2.8-3.0
Lard	—	0.934-0.938	36-40.5	27-30	1.1	195.4	50-70	93-96	24.0-27.5	3.7-4.1	0.5-1.3
Beef tallow	0.943-0.952	40-45	27-35	0.5	193-200	38-46	95.6	—	—	3.5-5.0	2.6-8.7
Mutton tallow	0.937-0.953	44-45	36-41	—	192-195	35-46	95.5	—	—	1.7-14.0	—
Butter	0.926-0.940	28-33	20-23	25.0-30.4	22.7	26-38	36.5-89.5	—	—	0.4-35.4	2.0-8.6
Sesame-oil	0.923-0.924	—	—	—	1.2	189-193	103-108	95.5	65	—	0.23
Olive-oil	0.916-0.918	—	—	± 6-+ 2	0.6	185-196	79-88	95	41-45	0.9-0.95	2-5.0
Rapese-oil	0.913-0.917	—	—	- 2- - 1.0	0.6	170-179	94-102	95.2	35-64	1.25-1.44	1.5-13.0
Peanut-oil	0.917-0.921	—	—	- 3-zero	—	190-196	83-100	95.7	45-51	1.05-1.37	1.3

yield a hard product with sulphur chloride, similar to elaidin, whilst animal fats do not; so that in this way vegetable oils may, to a certain extent, be detected in the presence of those derived from animals.

*Process.*—Five grammes of the oil or fat are mixed with an equal quantity of 2 c.c. of carbon bisulphide and sulphur chloride in a tared evaporating basin on the water-bath, and heated, with thorough mixing, until all volatile substances are expelled. The mass is washed free from oil on a filter with carbon bisulphide, dried and weighed, and its weight subtracted from the original weight of sample taken. The nature and consistency of the pasty or powdered mass is also observed, as various oils and fats yield different products.

## CHAPTER XI

### SUGAR AND SACCHARINE PRODUCTS

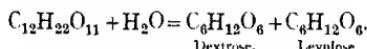
#### Classification of Sugars.

THE term "sugar" usually denotes cane or beet sugar (sucrose), but in its strictest sense is applied generically to a large class of compounds of the carbohydrate group, having for the most part the constitution of hexatomic alcohols ( $C_6H_{12}O_6$ , mannite), or of aldehydes or ethers, these latter bodies, although not true sugars, sometimes possessing sweet flavours and the characteristic reactions of the sugars.

SUGARS.		
HEXATOMIC ALCOHOLS ( $C_6H_{12}O_6$ ).	Glucoses ( $C_6H_{12}O_6$ ).	Saccharoses ( $C_{12}H_{22}O_{11}$ ).
Mannite, mannitol, dulcite, dulcitol, isodulcite, isodulcitol, sorbite, sorbitol.	Sucro-dextrose, Dextro-glucose, Dextrose, grape-sugar, Starch-sugar, Sucro-levulose, Levo-glucose, levulose, Galactose, arabinose, Pectinose, eucalyptose, Eucalyn, sorbinose, Sorbin, inosite, Scyllite.	Sucrose, saccharose, Cane-sugar, Lactose, lactin, Milk-sugar, maltose, Malt-sugar, mycose, Trehalose, melitose, Melitose, eucalyptin, Melezitose.

Sugars in foods are divided up into two main groups—saccharoses and glucoses—which are closely related. For example, by "inver-

sion" sucrose is converted by the action of heat or dilute acids into dextrose and levulose, both of which are glucoses.



Similarly, lactose is inverted into dextrose and galactose.

On the other hand, the hexatomic alcohols and the glucoses are only slightly affected by treatment with acids.

Glucoses and saccharoses rotate the vibratory plane of polarized light, and thus afford a means of estimating their amount present in solutions.

Hexatomic alcohols are unfermentable, saccharoses either unfermentable or only partially so with difficulty, while glucoses have a great tendency to ferment.

*Occurrence.*—Cane-sugar or sucrose occurs in the sugar-cane, beetroot, and in the juices of many plants—e.g., certain palms and sorghum—and in many fruits in combination with invert sugar (dextrose and levulose) in various amounts.

(Payen.)	Cane-Sugar.	Total Sugar.
Pineapple (Montserrat)	11.33	13.30
Strawberry (Collima d'Ehrherdt)	6.33	11.31
Apricot	6.04	8.78
Apple, grey Reinette (fresh)	5.28	14.00
Apple, grey Reinette (preserved)	3.20	15.83
Apple, English	2.19	7.65
Apple, Calville (preserved)	0.43	6.25
Plum, Mirabelle	5.24	8.67
Plum, Reine Claude	1.23	5.55
Orange	4.22	8.58
Lemon	0.41	1.47
Raspberry	2.01	7.23
Peach	0.92	1.99
Pear	0.68	8.78
Pear, St. Germaine (preserved)	0.36	7.84

**Cane-Sugar or Sucrose.**—This is the most commonly occurring of all the sugars. It crystallizes from supersaturated solutions in monoclinic prisms, generally possessing hemihedral faces. Its specific gravity is 1.595. It melts at 160° C., and on cooling does not recrystallize, but forms an amber-coloured solid. When heated to a temperature above 160° C., it decomposes into dextrose and levulosan—



At still higher temperatures it loses water, and is finally charred to caramel ( $C_{12}H_{18}O_9$ ) and other complex bodies.

Sucrose is very soluble in water, almost insoluble in absolute alcohol, and is insoluble in ether, chloroform, and many other anhydrous solvents.

The action of sucrose upon the ray of polarized light is very useful, the specific rotatory power of its 10 per cent. solution being  $\alpha_{D}66^{\circ}48'$  and  $\alpha_{D}73^{\circ}8'$ .

The sugar-cane is cultivated in many tropical countries, notably in the West Indies, Cuba, and the Southern States of America. The following is a table by Allen :—

Locality of Cane.	Water.	Sugar.	Woody Fibre.	Salts.	Authority.
Martinique ..	72·1	18·0	9·9	—	Pelligot.
Guadalupe ..	72·0	1·8	9·8	0·4	Dupuy.
Havana ? ..	77·0	12·0	11·0	—	Casaseca.
Cuba ..	65·9	17·7	16·4	—	Casaseca.*
Mauritius ..	69·0	20·0	10·0	1·0	Icery.
Ribbon cane ..	76·73	13·39	9·97	0·39	Avequin.
Tahiti ..	76·08	14·28	8·87	0·35	Avequin.

#### Average Composition of Ripe Cane Ash (Phipson).

Silica ..	..	..	..	..	..	48·0
Phosphoric acid ..	..	..	..	..	..	6·0
Sulphuric acid ..	..	..	..	..	..	8·0
Chlorine ..	..	..	..	..	..	4·5
Lime ..	..	..	..	..	..	10·0
Magnesia ..	..	..	..	..	..	6·5
Potash ..	..	..	..	..	..	18·0
Soda ..	..	..	..	..	..	2·0
Iron, alumina, etc. ..	..	..	..	..	..	2·0
						100·00

#### Average Composition of Raw Cane and Beet Sugar Ash (Monier).

	Cane-Sugar.	Beet-Sugar.
Potassium and sodium carbonates ..	16·5	82·2
Calcium carbonate ..	49·0	6·7
Potassium and sodium sulphates ..	16·0	11·1
Sodium chloride ..	9·0	Nil.
Silica and alumina ..	9·5	Nil.
	100·00	100·00

## Average Composition of Raw and Refined Sugar (Thorp).

	RAW SUGAR.				
	Cane-Sugar.	Glucose.	Water.	Organic Matter.	Ash.
Good centrifugal	.. 96·5	0·75	1·50	0·85	0·40
Poor centrifugal	.. 92·0	2·50	3·00	1·75	0·75
Good Muscovado	.. 91·0	2·25	5·00	1·10	0·65
Poor Muscovado	.. 82·0	7·00	6·00	3·50	1·50
Molasses sugar	.. 85·0	3·00	5·00	5·00	2·00
Jaggery sugar	.. 75·0	11·0	8·00	4·00	2·00
Vanilla sugar ..	.. 87·0	5·50	4·00	2·25	1·25
Beet-sugar (first)	.. 95·0	Nil.	2·00	1·75	1·25
Beet-sugar (second)	.. 91·0	0·25	3·00	3·25	2·50

	REFINED SUGAR.				
Granulated sugar	.. 99·8	0·20	Nil.	Nil.	Nil.
White coffee sugar	.. 91·0	2·40	5·50	0·80	0·30
Yellow XC sugar	.. 87·0	4·50	6·00	1·50	1·00
Yellow sugar ..	.. 82·0	7·50	6·00	2·50	2·00
Barrel sugar ..	.. 40·0	25·00	20·00	20·00	5·00

## Analysis of Sugar Beet (Payen).

Water ..	..	..	..	..	..	82·7
Sugar ..	..	..	..	..	..	11·8
Cellulose ..	..	..	..	..	..	0·8
Albuminous matter ..	..	..	..	..	..	1·5
Fatty matter ..	..	..	..	..	..	0·1
Pectin matter, phosphates, etc.	..	..	..	..	..	3·7
						100·1

**Maple-Sugar.**—This sugar is obtained by boiling down maple sap in open pans. It is frequently adulterated with molasses or cane-sugar, golden syrup and glucose.

The ash of pure maple-sugar varies from 0·35 to 0·40 per cent.

**Sorghum.**—The cultivation of sorghum, maize, and pearl millet, and the extraction of the sugar from their stalks, has been investigated by the U.S.A. Department of Agriculture, and is not used to any extent.

**Dextrose or Grape-Sugar.**—Dextrose ( $C_6H_{12}O_6 \cdot H_2O$ ), frequently called "glucose" or "starch-sugar," occurs in honey with levulose, and in various fruits, together with levulose and cane-sugar. It is obtained by the action of dilute acids on certain fer-

ments, upon starch, certain gums, dextrin, cane-sugar, and other vegetable matters.

Dextrose is less soluble and inferior in sweetening power to cane-sugar.

The specific rotatory power is  $\alpha_{D} 52^{\circ} 3^{\circ}$   $\alpha_{D} 58^{\circ}$ .

**Levulose** is sometimes called "fruit-sugar," and occurs along with dextrose in honey and in certain fruits. It is obtained by the action of dilute acids on inulin, and is usually in the form of a syrup which is capable of being crystallized only with great difficulty.

Levulose is sweeter than either dextrose or cane-sugar, and is more soluble than the former. The cupric-reducing powers of levulose and dextrose are accepted as being identical.

The specific rotatory power of levulose at  $15^{\circ}$  C. is  $\alpha_{D} - 98.8^{\circ}$ , decreasing by  $0.6385^{\circ}$  C. for each increase of  $1^{\circ}$  C. in temperature. At  $87.2^{\circ}$  C. its rotation is  $-52.7^{\circ}$ , being equivalent to, but opposite in direction to, that of dextrose at the same temperature.

**Milk-Sugar, Galactose, or Lactose.**—Lactose is formed from the whey of milk, after purification and concentration *in vacuo*.

Freshly prepared solutions of lactose exhibit a greater optical activity than after keeping. This action, which is known as "bi-rotation," is perhaps due to the presence of two modifications of bi-rotating sugars (isomeric glucoses)—(a) and (b)—one of which is ultimately converted into the latter either by standing for some time or upon heating.

The specific rotatory power of lactose after overcoming its bi-rotation is  $\alpha_{D} 52.5^{\circ}$ .

**Raffinose** ( $C_{18}H_{32}O_{16} \cdot 5H_2O$ ).—This sugar belongs to the class called saccharoids, or saturated alcohols, of which mannite is the type. It occurs in large quantities in Australian manna (certain eucalypti), in cotton-seeds, and in beetroot molasses to the extent of from 3 to 4 per cent., being more soluble than cane-sugar. Its crystals have peculiar terminal points, soluble in water, and possessing a slightly sweetish taste. It does not reduce Fehling's solution, readily undergoes fermentation, yielding upon hydrolysis fructose and melibiose ( $C_{12}H_{22}O_{11}$ ).

The specific rotatory power of raffinose at  $20^{\circ}$  C. is  $\alpha_{D} 104.5^{\circ}$ .

**Maltose or Malt-Sugar** occurs in fine crystalline needles,

and is a constituent of commercial glucose, being formed by the action of ptyaline in the process of digestion, and also when starch is acted upon by diastase at 55° to 60° C.

Freshly prepared solutions of maltose are bi-rotatory, which, contrary to lactose (*q.v.*), increase on standing.

The specific rotatory power of anhydrous maltose is  $\alpha_{D} 139.2^{\circ}$ ,  $\alpha_{I} 154.5^{\circ}$ .

**Commercial Glucose.**—Dextrin, often called "British gum," occurs in bread and in commercial glucose. It is an amorphous, colourless, tasteless substance, forming on hydrolysis with acid dextrose.

Its specific rotatory power is  $\alpha_{I} 200^{\circ}$ .

Commercial dextrin consists largely of erythro-dextrin, yielding a brown coloration with iodine. Pure dextrin is not coloured by iodine, and has no cupric-reducing power.

Commercial glucose, grape-sugar, starch-sugar, mixing or crystal syrup, is largely used by brewers and preserve manufacturers.

Starch glucose is manufactured in France and Germany from potato starch, and in America from maize starch, by boiling with a 1 to 3 per cent. solution of sulphuric acid, the residual acid being subsequently eliminated by neutralization with lime or chalk. The resulting clear solution is filtered through bone black, and concentrated either to a thick, almost colourless syrup or to a solid mass.

			Percentage of Syrup.	Percentage of Solid.
Dextrin	..	..	29.0 to 45.0	0.0 to 9.00
Maltose	..	..	4.0 to 19.0	0.0 to 2.00
Dextrose	..	..	34.0 to 36.0	70.0 to 99.5
Ash ..	..	..	0.3 to 0.6	0.3 to 0.75
Water	..	..	14.0 to 17.0	0.6 to 17.00

### The Polarimeter and Saccharimetry.

Most sugars alter the plane of polarization of a ray of light, not only in their solid condition, but also in solution, the degree of rotation being directly proportional to the concentration.

For a complete description of polariscopes and polarimetry the reader is referred to the special text-books by Landolt, Rolffé, and others.

The form of instrument most in use in this country and in the U.S.A. is that of the Soleil-Ventzke, provided either with a double or triple shade device, the best-known makers being Messrs. Schmidt and Haensch, and Julius Peters, Berlin—the instruments of the latter maker differing in the main from that of the former in the mounting, the latest instruments being fitted with Lippich prisms, the optical parts being enclosed in dustproof boxes, which largely mitigate the effects of sudden changes of temperature.

The normal weight (number of grammes substance in 100 c.c. solution) used in the Soleil-Ventzke instruments is 26·048 grammes.

**Specific Rotatory Power.**—This is a term used to express the comparative optical activity of various substances, and is the angular degree or degrees through which the plane of polarization of a ray of light of known wave-length is rotated in traversing a thickness of 1 decimetre of substance, prepared by dissolving 1 gramme in 1 c.c. water in a column 1 decimetre in length.

The "absolute" specific rotatory power is that of a pure solid. When this is unattainable, that of its solution is known as the "sensible" or "apparent" specific rotatory power.

The actual rotatory power of a solution varies directly with the length of the column traversed by the ray, with the degree of concentration, and with the wave-length of the light.

The specific rotatory power is expressed as  $a_D$ , or  $a_j$ , according to the character of the light. D indicates the monochromatic or sodium flame, and  $j$  the transition tint or complementary to the yellow light.

The specific rotatory power is found as follows :

$$a_D \text{ or } a_j = \frac{100}{cl} a,$$

Where  $a$  equals the degree of angular rotation observed;

Where  $l$  equals the length of tube in decimetres;

Where  $c$  equals the degree of concentration in grammes per 100 c.c. of the fluid.

**Preparation of Solution for the Polarimeter.**—Mix the sample thoroughly, and weigh out as rapidly as possible the standard quantity (if the Soleil-Ventzke is used, this will be 26·048 grammes)

in the tared German-silver dish, weighing to 0·005 grammie, and introduce it into a 100-c.c. flask. It is absurd to weigh out the normal weight to milligrammes, as it is impossible to read the meniscus of the solution in a 100-c.c. flask to less than a centigramme—that is, equal to an error of 0·01 per cent.—and in endeavouring to weigh more accurately some substances are so very hygroscopic that the time thus involved entails even a greater error. The official German method is to wash the substance into the flask until

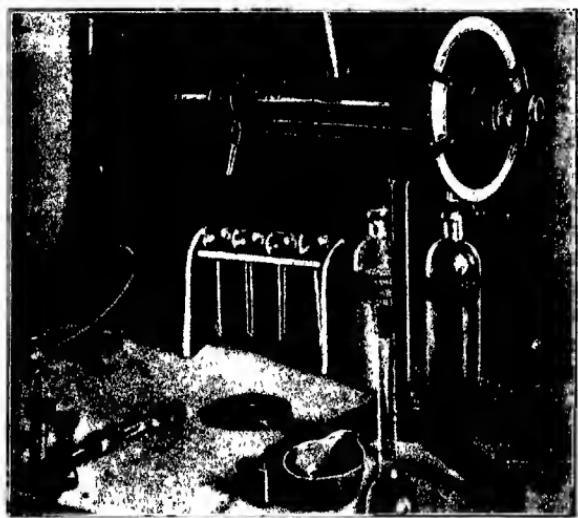


FIG. 34.—POLARISCOPE AND ACCESSORIES.

the flask is three-quarters full, and shake until the contents are all dissolved. The flask is then filled up to the mark at the required temperature and well shaken up.

Many aqueous solutions are opaque and require clarification. This is done by adding usually about 2 c.c. of a solution of basic lead acetate of specific gravity 1·25 before making up to the mark. Any foam may be dispelled by adding 1 drop of ether to the top of the solution. After thorough admixture, the solution is filtered through a dry filter into a dry cylinder, rejecting the first few drops.

The cylinder is covered with a glass to prevent evaporation. The nature and amount of the clarifier used depends on the class of sugar under examination. The only rule is : Use as little clarifier as possible. If too little lead is used, the clarification is incomplete ; if too much, the solution becomes cloudy after filtration ; if the proper quantity has been added, a rapid coagulation and settling will take place, leaving a clear solution. A very opaque solution should not require more than about 10 c.c. lead. The use of lead should be followed up by the addition of a smaller quantity of cream of alumina, as the presence of the former salt tends to increase the readings. A 5 per cent. solution of common salt also answers the purpose very well. The French plan of treatment with animal charcoal is objectionable, owing to the absorption of a certain amount of the sugar. This may be rectified to a very great extent by merely filtering through a layer of the charcoal and rejecting the first runnings as being weak in sugar content. Carefully avoid all air-bubbles in the observation tube and undue pressure of the cover-glasses, as the strain sets up an unequal distribution of density, and thus causes a rotating effect on the light rays. The field should be as evenly and brightly illuminated as possible, and sharply focussed.

#### Estimation of Sucrose in Presence of Glucose.

Sucrose is inverted by the action of dilute acids, invertase or yeast, into the two sugars, dextrose and levulose, in equal proportions, the resulting product being termed invert sugar.

The rotatory power of levulose, unlike that of sucrose and dextrose, is dependent upon the temperature, so that, as the specific rotation of levulose and dextrose are assumed as equal, a solution of invert sugar at  $87\cdot2^{\circ}$  C. polarizes at zero, whilst, as the temperature decreases from  $87\cdot2^{\circ}$  C., the rotatory power of the levulose increases, till at  $0^{\circ}$  C. a normal solution of invert sugar would polarize at  $-44^{\circ}$ .

Where S equals the per cent. sugar,  $\alpha$  equals the direct polarization,  $b$  equals the invert polarization, and  $t$  equals the temperature, the formula worked out by Clerget for obtaining the percentage of sucrose present in a mixture of sucrose and invert sugar,

based upon the rotation of cane-sugar before and after inversion, is—

$$S = \frac{100(a - b)}{144 - \frac{b}{2}},$$

assuming that a normal solution of sucrose polarizes at  $100^\circ$  before inversion and  $-44^\circ$  after inversion, decreasing  $0.5^\circ$  for each degree in temperature above  $0^\circ\text{C}$ .

The process of inversion usually causes an increase in bulk in the solution. This increase is corrected by using a 22-centimetre tube for the invert readings instead of a 20-centimetre, as in the case of the direct polarization. This inversion tube, as it is called, is provided with a short vertical thermometer tube, and is water-jacketed, so that a constant temperature may be sustained throughout the period of readings.

**Process of Inversion.**—Fifty c.c. of the clarified solution used in the direct polarization are placed in a 50-c.c. flask, 5 c.c. concentrated hydrochloric acid added, and the whole well mixed. It is convenient for this purpose to have a flask graduated both at 50 c.c. and at 55 c.c. The flask and contents are then heated on the water-bath in such a way that in the course of ten minutes the temperature will have risen to  $68^\circ\text{C}$ . The solution having been kept at this temperature for five minutes, is then suddenly cooled to that of the laboratory, decolourized if necessary with animal charcoal, transferred to the 22-centimetre tube, and polarized. If a 20-centimetre tube be employed, 10 per cent. must be added to the reading.

Molasses and other dark-coloured products may be observed in a 100 or 110 centimetre tube, should the solution be too dark, and the reading multiplied by 2.

When extreme accuracy is required, however, Wiley's double dilution method should be employed, which makes allowance for the large volume of precipitate obtained in clarification.

For this purpose a half-normal weight of the sample, after clarification, is made up to 100 c.c., as well as a normal weight made up to the same volume. The direct and invert readings are taken in both instances, the true reading being that of the product of the half-normal and normal readings divided by their difference in either direct or invert polarizations.

Molasses, etc., are frequently adulterated with commercial glucose, the presence of which is indicated by an extra high direct, together with a dextroinvert, polarization.

The optical activity of commercial glucose is due to the presence of dextro-maltose and dextrose, together with small amounts of higher saccharine compounds.

An approximate estimate of the amount of this substance present in a sample of molasses, etc., may be obtained by the use of the formula—

$$G = \frac{(\alpha - S) 100}{175},$$

where G equals the per cent. common glucose,  $\alpha$  the direct polarization, and S the per cent. sucrose obtained by Clerget's formula.

A more accurate and perhaps easier method is that of Chandler and Rickett. Invert sugar, as has already been mentioned, is optically inactive at 87·2° C., owing to the neutralizing effect of the levulose upon the dextrose rotation, while the rotatory power of starch-sugar is unaltered by increase in temperature.

If the prepared solution be, therefore, polarized at this temperature, the specific rotation of the starch-sugar alone will be observed. Unfortunately, the exact estimation of commercial glucose is impossible, owing to its varying composition.

### Estimation of Sugars by Cupric Reduction.

All common sugars, with the exception of sucrose, possess the power of reducing alkaline cupric solutions.

Of the varied solutions, that of Fehling, which is an alkaline tartrate solution of cupric sulphate, and has many modifications in formula and use, is the most commonly used. It is prepared in two stock solutions, the mixture of equal parts of which produce the reagent.

1. *Copper Solution*.—34·639 grammes pure recrystallized copper sulphate dissolved in distilled water and diluted to 500 c.c.

2. *Alkaline Tartrate Solution*.—Seventy grammes pure caustic soda and 180 grammes recrystallized Rochelle salt dissolved in distilled water and diluted to 500 c.c.

The solutions are mixed as required, are frequently tested as to

their freshness by diluting with an equal quantity of distilled water, and boiled with it for a few minutes, when they ought to remain clear, and are standardized against a 0·5 per cent. aqueous solution of pure anhydrous glucose, 10 c.c. of which should exactly reduce 10 c.c. Fehling's solution. In the estimation of reducing sugars by Fehling's solution it is necessary to neutralize all free acid in the saccharine liquid, and in volumetric determinations the latter should be as clear and colourless as possible.

#### Volumetric Estimation by Fehling's Solution.

The sugar content of the liquid should not be more than 0·5 to 1 per cent. This can easily be arranged by carrying out a rough preliminary assay.

Measure out exactly, by means of a pipette, 10 c.c. Fehling's solution into a small flask, add 30 c.c. water (the amount of dilution should be as constant as possible), and heat to boiling over a Bunsen burner. While boiling, run in from a burette the sugar solution, 2 c.c. at a time, boiling for an equal period (usually two minutes by stop-watch) after each addition.

As the colour disappears, smaller quantities of the saccharine liquid must be added, until after two minutes' boiling the copper is all reduced to cuprous oxide, leaving a colourless or only faintly yellow supernatant fluid. If all the copper is not reduced, a faint blue film will be observed upon holding the flask up to a brightly lit window. The exact end-point is ascertained by filtering a few drops of the supernatant liquid, and testing with acetic acid and potassium ferrocyanide, when a more or less reddish-brown precipitate or coloration will indicate unreduced copper. A better indicator, which does not require filtration, is prepared by boiling 0·05 grammes starch with a few c.c. of water, adding 10 grammes KI, and diluting to 100 c.c. About 0·6 or 1 c.c. of this solution is taken, acidified with a drop or two of acetic acid, and one drop or more of the unfiltered assay solution added (upon a clean white tile). A colour varying from red to blue will be produced as long as any unreduced copper is present. The end-point is observed by the absence of any coloration. This solution does not keep, as a rule, more than a few hours.

The titration should then be repeated by running in almost all the sugar solution necessary into the boiling Fehling, and boiling for a period of two minutes after each addition until a variation of not more than 0·1 c.c. sugar solution is obtained.

The following are the equivalents of Fehling's solution :

$$10 \text{ c.c. Fehling's solution} = \begin{cases} 0\cdot0500 \text{ gramme dextrose, levulose, or invert sugar.} \\ 0\cdot0475 \text{ gramme cane-sugar (after inversion).} \\ 0\cdot0678 \text{ gramme lactose.} \\ 0\cdot0807 \text{ gramme maltose.} \end{cases}$$

In estimating sucrose by Fehling's solution the solution is inverted in the usual way (*vide* Polarimetric Analysis) with 5 c.c. acid, carefully neutralized with caustic soda, and diluted to 0·5 or 1 per cent. strength before titration.

#### Gravimetric Estimation by Fehling's Solution.

Sixty c.c. Fehling's solution are mixed with 60 c.c. water in a beaker, and heated by placing the beaker in boiling water. Twenty-five c.c. of the dilute sugar solution are then added, and the whole replaced in the hot water for ten minutes, care being taken to ensure excess of Fehling. The liquid is then rapidly filtered through a Gooch-filter, washed with boiling water, dried, and weighed either as Cu<sub>2</sub>O, or, better, strongly ignited and weighed as CuO.

	Glucose.	Cane-Sugar (after Inversion).	Lactose.	Maltose.
Cu ..	0·5634	0·5395	0·7707	0·9089
Cu <sub>2</sub> O ..	0·5042	0·4790	0·6843	0·8132
CuO ..	0·4535	0·4308	0·6153	0·7314

#### Volumetric Estimation by Pavy-Fehling's Solution.

This process, which is dependent upon the fact that in a strongly ammoniacal solution the cuprous oxide is held in a colourless solution, is an extremely useful method for estimating sugars in admixture with other substances which require a more or less tedious process of preliminary separation—e.g., milk and beetworts.

**Preparation of Solution.**—One hundred and twenty c.c. Fehling solution are diluted with 300 c.c. ammonia (specific gravity, 1·880) and 400 c.c. caustic soda (specific gravity, 1·14; equal to 12 per cent.), and the liquid made up to 1,000 c.c.

Then 100 c.c. Pavv will equal 0.050 grammie glucose (equal to 10 c.c. Fehling).

**Process.**—One hundred c.c. of the cupric solution are placed in a flask, and the dilute sugar solution run in while boiling from a burette in the ordinary way. The reaction is somewhat slower than with the Fehling test, but the end-point is very sharp.

## CHAPTER XII

### TEA, COFFEE, AND COCOA

#### Tea.

TEA is the dry leaf of the species *Thea* of the genus *Camellia*, the appearance of which, whether black or green, depends upon the mode of preparation. The crop is gathered four times each season, the leaves being assorted according to their age, the finest teas being obtained from the youngest leaves from young shrubs. Formerly "green teas" were much in demand, amongst them being Capers, Gunpowder, and Hyson. Caper teas are, however, more or less adulterated, as a rule. They are dried by roasting shortly after collecting. To obtain black tea, the leaves are allowed to partially ferment by remaining in heaps for about twelve hours, when, after further treatment, they are gradually roasted over open charcoal fires. To this class belong Assam, Indian, Ceylon, and Japanese, Kaison, Moning, and Oolong.

The constituents of tea are essential oil, theine, quercitin, tannin, boheic acid, gallic acid, oxalic acid, querci-tannic acid, chlorophyll, gum, resin, wax, substances of albuminous, fibrous, and colouring nature, and mineral matter or ash.

Tea may be adulterated by the addition of exhausted leaves, or the leaves of the willow, sloe, etc., from which it may be distinguished by microscopical examination. For this purpose the leaf to be examined may be prepared in two ways: First, place the portion between two glasses, weighing them with a piece of silver, and then burn the leaf carefully over a thin sheet of either silver or platinum. Secondly, after securing the leaf as before between two cover-glasses with a suitable weight, heat gently in a strong alkaline solution of potassium permanganate, taking care not to overdo the action. After all organic matter except the skeleton

structure has been destroyed, the leaf may be carefully washed by rinsing in water and hydrochloric acid to clear it, and examined microscopically.

Tea is frequently mixed with mineral matter—*e.g.*, sand, magnetic iron ore, quartz, mica, and the like—notably Capers and Congou.

For strengthening the colour of the extract and its astringency, catechu and alkalies are added.

Teas are also faced, particularly green teas, by mixing with indigo, turmeric, graphite, etc.

The following is the mean analysis of sixteen teas by König :

Moisture	..	..	..	..	11.49
Nitrogenous substances	..	..	..	..	21.22
Theine	..	..	..	..	1.35
Ethereal oil	..	..	..	..	0.67
Fats and waxes	..	..	..	..	3.62
Gums	..	..	..	..	7.13
Tannin	..	..	..	..	12.36
Other free nitrogenous matter	..	..	..	..	16.75
Fibre	..	..	..	..	20.30
Ash	..	..	..	..	5.11
Total	..	..	..	..	100.00

**Moisture.**—This is estimated by drying between tared watch-glasses at 100° C.

**Theine.**—Six grammes of finely powdered leaves are boiled under a reflux condenser with 500 c.c. water for about six hours, filtered, and washed with water till the filtrate bulks about 600 c.c. Heat to almost boiling, add about 4 grammes lead acetate, and boil for ten minutes under the reflux condenser. Enough lead must be added to effect complete precipitation, leaving a colourless supernatant layer. Pass through a dry filter. Evaporate 500 c.c. (equal to 5 grammes tea) down to 50 c.c., precipitate all excess of lead by the addition of a little sodium phosphate, filter, wash, concentrate filtrate and washings to about 40 c.c., and extract the alkaloid by six repeated treatments with chloroform in a separating-funnel. Place the chloroform solutions in a tared flask, evaporate off all traces of chloroform on the water-bath, and weigh the remaining alkaloid.

**Nitrogen.**—This is estimated by Kjeldahl's method.

**Extract.**—Two grammes of the finely powdered tea are boiled

for one hour in 100 c.c. water, filtered hot, and the residue boiled repeatedly with 50 c.c. water till all colouring matter is extracted. The cooled combined extractions after filtration are made up to a fixed volume, an aliquot part taken, evaporated to dryness, and weighed. The residue dried and weighed yields the insoluble matter.

**Tannin.**—This may be rapidly estimated by titrating the tannin-containing solution by a solution of lead acetate (0·5 per cent.), using an ammoniacal solution of potassium ferrocyanide as an indicator (5 milligrammes  $K_4FeCy_6$  and 5 c.c.  $NH_4OH$  in 5 c.c.  $H_2O$ ). The lead solution is standardized against a 0·1 per cent. solution of pure tannin. The tannin may also be estimated by Löwenthal's process.

**Ash.**—The total ash is obtained by gently igniting 1 to 5 grammes in a platinum dish. The residue is extracted with water in order to ascertain the soluble and insoluble.

**Gum.**—An aqueous extract is concentrated to a paste, extracted with methylated spirit, filtered, washed, and the gum extracted from off the filter with hot water, evaporated to dryness in a tared dish, and weighed.

#### Coffee.

Coffee consists of the dried and toasted seeds of the *Coffea Arabica*, of the order Cinchonaceæ, and is obtained from India, Ceylon, Java, Arabia ("Mocha"), and Brazil.

The main constituents of coffee are : essential oil, concrete oil, theine or caffeine, an alkaloid "caccarine," caffeo-tannic acid, and manno-arabinose.

The following are analyses of coffees by Bell :—

	"MOCHA" COFFEE.		EAST INDIA COFFEE.	
	Raw.	Roasted.	Raw.	Roasted
Moisture .. .. .. ..	8·98	0·63	9·64	1·13
Caffeine .. .. .. ..	1·08	0·82	1·11	1·05
Saccharine matter .. .. .. ..	9·55	0·43	8·90	0·41
Caffeic acids .. .. .. ..	8·46	4·74	9·58	4·52
Alcoholic extract, containing nitrogenous and colouring matter ..	6·90	14·14	4·31	12·67
Fat and oil .. .. .. ..	12·60	13·59	11·81	13·41
Legumin and albumin .. .. .. ..	9·87	11·23	11·23	13·13
Dextrin .. .. .. ..	0·87	1·24	0·84	1·38
Cellulose and insoluble matter ..	37·95	48·62	38·60	47·42
Ash .. .. .. ..	3·74	4·56	3·98	4·88
	100·00	100·00	100·00	100·00

**Specific Gravity.**—This determination shows whether the berries have been artificially moistened or not. König found, for example, that coffee roasted with glucose syrup caused an increase of 7 per cent. of moisture. The specific gravity of genuine raw coffee ranges from 1·368 to 1·041, and that of roasted berries from 0·635 to 0·500. The specific gravity may be determined either in Regnault's volumenometer modified by Dupré, or by filling a 50-c.c. flask with the berries up to the graduation mark, noting the weight, and again noting the weight when the flask is filled with mercury to the same mark, the increase in weight being due to the mercury filling the spaces between the coffee berries.

$$\text{Weight of berries in grammes} \times 13\cdot50 \\ 50 \text{ c.c.} \times 13\cdot59 - \text{weight of interstitial Hg.} \} = \begin{array}{l} \text{Specific gravity of} \\ \text{the berries.} \end{array}$$

The other constituents are determined as in tea.

Fictitious coffee is formed from chicory, bran, flour, seeds of various kinds, date-stones, etc. These are observed on microscopic examination. A. Franz states that a 10 per cent. infusion of coffee in boiling water, when treated with a 2·5 per cent. solution of copper acetate, yields on filtering a greenish-yellow solution, an infusion containing chicory yielding under similar treatment a dark brown filtrate, changing on standing.

**Ash.**—Pure coffee yields a white ash of from 3·5 to 5·0 per cent.

#### Cocoa and Chocolate.

Cocoa is the seed of the *Theobroma cacao*, of the order Byttneriaceæ, existing in Central America, South America, West and East Indies, etc. The fruit contains twenty-five to forty ovate, flattened seeds, covered with a thin, brittle shell. The seeds are fermented before separation, and then slowly dried, the flavour and aroma being altered from a disagreeable to that of a pleasant one, largely owing to the method of manipulation. The dried seeds are exported, roasted, separated from the shell, and ground.

The following are the average analyses of eight samples by König :

	Moisture.	Nitrogenous Matter.	Oil.	Starch.	Cellulose.	Ash.
Cocoa bean (shell-free)	3·25	14·76	49·08	13·31	3·68	3·65
Cocoa husks ..	7·83	14·29	6·38	—	14·69	7·12

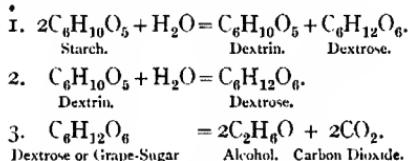
Cocoa may be adulterated with sugar, starches, Venetian red, brick-dust, peroxide of iron, ochre, and various pigments.

## CHAPTER XIII

### ALCOHOL AND FERMENTED BEVERAGES

#### Formation of Alcohol.

ALCOHOL is formed by the spontaneous fermentation of grape-sugar. Grape-sugar or dextrose is formed within plants by the saccharous fermentation of starch and dextrin :



These equations represent theoretically the conversion of a starch into alcohol and carbon dioxide, showing no by-products. The actual yield, however, is about 95 per cent. of the above, together with about 5 per cent. of succinic acid, glycerin, higher alcohols and ethers (or esters), and traces of other complex substances.

Alcoholic products may be divided into two classes : (1) Distilled or potable spirits ; (2) Fermented liquors. The latter class is subdivided into : (a) Malted and brewed liquors—*e.g.*, beer; and (b) Directly fermented (spontaneous) saccharine materials or juices—*e.g.*, wines.

#### Analysis of Alcoholic Liquids.

**Specific Gravity.**—This is determined by any of the usual methods at  $15^{\circ}\text{C}$ .

**Estimation of Alcohol—By Specific Gravity.**—Liquors which leave little or no extractive matter upon evaporation may be, for approximation, considered as containing only alcohol and water.

The specific gravity is therefore applied direct to the alcohol table.

*By Evaporation.*—The specific gravity having been taken, a quantity of the sample (50 to 100 c.c.) is concentrated to 25 per cent. its original volume in an open vessel, the residue being made up to the original volume. The specific gravity of this

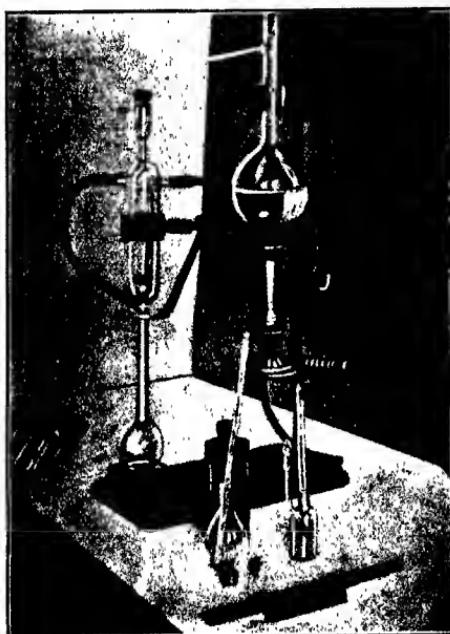


FIG. 35.—DISTILLATION AND ESTIMATION OF ALCOHOL WITH SPECIFIC-GRAVITY BOTTLES.

solution is now taken, and subtracted from that of the original specific gravity + 1. E.g., the specific gravity of sample was 0·8552, and specific gravity of residual solution (after making up to bulk at same temperature) 0·9263; then  $1\cdot8552 - 0\cdot9263 = 0\cdot9289 = 52\cdot82$  per cent. absolute alcohol (by volume).

*By Distillation.*—A suitable quantity (usually 100 c.c.) of the

sample is measured at  $15.5^{\circ}$  C. in a graduated flask, neutralized with alkali, and distilled into the same flask until the residue occupies not more than 25 per cent. of the original volume in the case of beer and light wines, etc., and almost to dryness in the case of highly alcoholic liquors, as brandy and whisky.

The whole apparatus should be made of good glass, the condenser preferably being of the upright worm or American double-surface condensing patterns.

The receiver should be plugged loosely with cotton-wool, and kept cool in warm weather in iced water.

The distillate, having been made up to the original volume with distilled water at  $15.5^{\circ}$  C., is well mixed, and its specific gravity taken at that temperature. The percentage of alcohol present in the sample is obtained by reference to the alcohol tables.

"Proof spirit," by Act of Parliament, is defined as that of a liquid having a specific gravity of 0.91984 at  $15.5^{\circ}$  C., corresponding, therefore, to 49.24 per cent. by weight, or 57.06 per cent. by volume, of absolute alcohol. If higher in strength, it is said to be "over-proof"; if lower, then "under-proof." E.g., a liquor having 90 per cent. by volume of alcohol and 10 per cent. water would be termed "10 degrees under proof."

**Extract.**—This is estimated by evaporating a measured volume to dryness, and weighing the residue or "extract." Highly saccharine liquors, owing to dehydration of the sugars, should be dried at a temperature not exceeding  $75^{\circ}$  C. *in vacuo*.

**Ash.**—The residue or extract is ignited at a low red heat till completely white, and weighed when cool.

#### DISTILLED OR POTABLE LIQUORS.

The distillate from an alcoholic infusion or mash varies in its character and composition not only in accordance with the process in use, whether in a pot or patent still, but with the rate of distillation, state of rectification, and class of materials employed. The simpler form of old copper still and worm, known as the "pot-still," used in the manufacture of whisky, brandy, and rum, yields a very impure spirit, having a high content of secondary products, which impart to it more or less harshness.

Such characteristics may be overcome by maturing in casks or partial rectification. The product of the "patent," or what is commonly termed "Coffey," still is much less flavoured, and consists very largely of pure (ethyl) alcohol or "silent spirit." This featureless article does not require maturing, and is flavoured with more or less pot-still spirit or artificial chemical flavourings, composed of the higher alcohols, aldehydes, and esters. It is now possible to obtain spirit of 95 per cent. alcohol in two distillations. The results of analyses of the secondary products or *impuretés* are usually expressed in parts or grammes per 100,000 of absolute alcohol.

#### METHODS OF ANALYSIS.

All corks should be covered with tinfoil. Two hundred c.c. of the sample should be distilled down to 20 c.c., the distillate made up to the original volume at 15·5° C., and the specific gravity determined in the usual manner, from which the alcoholic content is ascertained. In this distillate the aldehydes, furfural, higher alcohols, and ethers are determined.

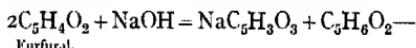
**Volatile and Fixed Acidity.**—The total acidity is first determined by titrating 25 c.c. of the sample with  $\frac{1}{10}$  soda. Another 25 c.c. is evaporated several times to dryness upon the water-bath, and also titrated with  $\frac{1}{10}$  soda, using phenolphthalein as indicator. The latter number of c.c.'s used multiplied by 0·0075 gives the fixed acidity expressed as tartaric acid, and the difference of amount in c.c.'s between the first and second titration multiplied by 0·006 shows the volatile acidity as acetic acid.

**Aldehydes.**—Dissolve 0·15 gramme fuchsine in 150 c.c. water, and add to it 100 c.c. of an aqueous solution of sodium bisulphite (specific gravity, 1·360) and also 15 c.c. of 66 per cent. sulphuric acid. Shake up well, and allow to stand till colourless. Also prepare a 1 per cent. solution by weight of pure acetic aldehyde in 50 per cent. alcohol.

Place 10 c.c. of the above-mentioned alcoholic distillate in a graduated cylinder (50 c.c. size), add 4 c.c. fuchsine solution, and make up to 20 c.c. with 50 per cent. alcohol. Also treat 5 c.c. of the standard aldehyde solution in a similar manner, and compare the tints after the lapse of twenty minutes in the usual colorimetric way (*vide* Estimation of Ammonia in Water, Nessler solution).

**Higher Alcohols.**—One hundred c.c. of the distillate to be tested are brought to 50 per cent. strength, placed in a 250-c.c. distillation-flask, and 2 to 3 grammes chlorohydrate of meta-phenylendiamine or phosphate of aniline (1 c.c. pure aniline and 1 c.c. phosphoric acid, specific gravity 1·453) are added, together with a piece of platinum foil to prevent bumping, and the liquid boiled gently under a reflux condenser for an hour, cooled, and then distilled, the time occupied in distillation being limited to forty minutes. The distillate is received into a 75-c.c. graduated flask, so that after shaking it will be of 66·7 per cent. strength. Ten c.c. of this distillate are pipetted into a 100-c.c. dry flask, and 10 c.c. monohydrated colourless sulphuric acid added with caution. After shaking, the mixture is allowed to remain at 120° C. for an hour in a calcium chloride water-bath. A standard comparison solution of the same (66·7 per cent.) alcoholic strength is prepared by dissolving 0·667 gramme pure isobutylic alcohol in a litre of 66·7 per cent. alcohol, and is likewise simultaneously treated with the acid for an hour at 120° C. The tints thus obtained are compared in the colorimeter.

**Compound Ethers.**—The ethers in brandy and whisky are mainly butyric, caproic, caprylic, and capric, and are saponified by standard alkali. Aldehydes and furfural are also acted upon by the alkali—



and must therefore be removed by boiling under a reflux condenser with phosphate of aniline (already described) for two hours previous to distillation.

One hundred c.c. of the purified distillate is neutralized with  $\frac{N}{10}$  soda (phenolphthalein indicator), and 25 c.c.  $\frac{N}{10}$  soda added in excess, and boiled gently under a reflux condenser for an hour, after which the excess of alkali is titrated with  $\frac{N}{10}$  hydrochloric acid in the cold. Each c.c. of difference between 25 c.c. and the final titration represents 0·0088 gramme ethyl acetate.

**Furfural.**—Furfural is usually absent, or only present in small proportions, in patent spirits, but is characteristic of pot-still products. It strikes a red colour with "aniline acetate [ $\text{C}_4\text{H}_3\text{O.CH(C}_6\text{H}_5\text{Me}_2\text{NH}_2)_2$ ].

A standard solution of furfural is prepared containing 0·005 grammes per litre.

Ten c.c. of this solution and 10 c.c. of the alcoholic distillate, reduced to 50 per cent. alcoholic strength, are each placed in a graduated cylinder, and 10 drops pure aniline added to each, and then simultaneously 1 c.c. pure (furfural-free) acetic acid added. The tints are compared after fifteen minutes.

#### MALTED LIQUORS (BEER, ETC.).

The analysis of beer usually consists of—

1. The alcohol.
2. Carbonic acid.
3. Volatile and fixed acids.
4. The percentage of malt extract (its composition) and original gravity.
5. Hop resin and glycerin.
6. Nature of bitter used.
7. The ash (including phosphates and chlorides).

**Alcohol.**—This is estimated by distillation as already described. It may also be estimated by boiling off the alcohol and carbonic acid, calculating the percentage of alcohol from the specific gravity before and after boiling (Tabarie's method), as has been described.

**Carbonic Acid.**—The absorbed gases may be drawn away from a measured quantity of the sample contained in a flask by means of a mercury pump. A convenient method is to expel all the carbon dioxide from a measured quantity of the sample in a flask connected with a sealed flask containing dilute ammonia in such a way that all the gas is absorbed by the ammonia. If the requisite quantity of calcium chloride be then added, and the contents of the second flask boiled till free from ammonia, the calcium will be precipitated, and may be filtered, washed, dissolved in a little standard acid, and titrated back with alkali (using cochineal indicator).

Two c.c.  $\frac{N}{10}$  acid = 10 milligrammes  $\text{CaCO}_3$  or 4·4 milligrammes  $\text{CO}_2$ .

**Volatile and Fixed Acids.**—The volatile (acetic) acid is obtained by distilling the sample almost to dryness and titrating the distillate thus obtained with  $\frac{N}{10}$  alkali. The fixed acid or acids

(usually lactic) are determined by titrating the residue in the flask after diluting with water (1 c.c.  $\frac{N}{10}$  alkali = 9 milligrammes lactic acid).

**Malt Extract.**—Five grammes of the sample are evaporated to dryness in a platinum dish on the water-bath, and the residue or extract thoroughly dried.

To ascertain the amount of malt originally used in the preparation of the beer, the percentage of alcohol, volatile acid (as acetic), and malt extract having been found, the "spirit indication" and "original gravity" must be calculated.

The "spirit indication" is obtained by subtracting the specific gravity of the alcoholic distillate from 1,000. The degrees of gravity lost are obtained from either of the following tables, dependent upon which process has been employed for estimating the alcohol. The degrees of gravity added to the specific gravity of the boiled beer yield "the original gravity of the wort." From the original gravity the malt extract is obtained by reference to the malt extract table.

TABLE A.

#### **IF THE DISTILLATION PROCESS IS USED.**

TABLE B.  
IF THE EVAPORATION PROCESS IS USED.

Degrees of Spirit Indication.	o.o.	o.1.	o.2.	o.3.	o.4.	o.5.	o.6.	o.7.	o.8.	o.9.
0	0'0	0'3	0'7	1'0	1'4	1'7	2'1	2'4	2'8	3'1
1	3'5	3'8	4'2	4'6	5'0	5'4	5'8	6'2	6'6	7'0
2	7'4	7'8	8'2	8'7	9'1	9'5	9'9	10'3	10'7	11'1
3	11'5	11'9	12'4	12'8	13'2	13'6	14'0	14'4	14'8	15'3
4	15'8	16'2	16'6	17'0	17'4	17'9	18'4	18'8	19'3	19'8
5	20'3	20'7	21'2	21'6	22'1	22'5	23'0	23'4	23'9	24'3
6	24'8	25'2	25'6	26'1	26'6	27'0	27'5	28'0	28'5	29'0
7	29'5	30'0	30'4	30'9	31'3	31'8	32'3	32'8	33'3	33'8
8	34'3	34'9	35'5	36'0	36'6	37'1	37'7	38'3	38'8	39'4
9	40'0	40'5	41'0	41'5	42'0	42'5	43'0	43'5	44'0	44'4
10	44'0	45'4	46'0	46'5	47'1	47'6	48'2	48'7	49'3	49'3
11	50'3	50'9	51'4	51'9	52'5	53'0	53'5	54'0	54'5	55'0
12	55'6	56'2	56'7	57'3	57'8	58'3	58'9	59'4	59'9	60'5
13	61'0	61'6	62'1	62'7	63'2	63'8	64'3	64'9	65'4	66'0
14	66'5	67'0	67'6	68'1	68'7	69'2	69'8	70'4	70'9	71'4
15	72'0	—	—	—	—	—	—	—	—	—

TABLE C.  
FOR ASCERTAINING THE VALUE OF ACETIC ACID.

Excess per Cent. of Acetic Acid in the Beer.	Corresponding Degrees of Spirit Indication.									
	o.00	o.01	o.02	o.03	o.04	o.05	o.06	o.07	o.08	o.09
0'0	—	0'02	0'04	0'06	0'07	0'08	0'09	0'11	0'12	0'13
0'1	0'14	0'15	0'17	0'18	0'19	0'21	0'22	0'23	0'24	0'26
0'2	0'27	0'28	0'29	0'31	0'32	0'33	0'34	0'35	0'37	0'38
0'3	0'39	0'40	0'42	0'43	0'44	0'46	0'47	0'48	0'49	0'51
0'4	0'52	0'53	0'55	0'56	0'57	0'59	0'60	0'61	0'62	0'64
0'5	0'65	0'66	0'67	0'69	0'70	0'71	0'72	0'73	0'75	0'76
0'6	0'77	0'78	0'80	0'81	0'82	0'84	0'85	0'86	0'87	0'89
0'7	0'90	0'91	0'93	0'94	0'95	0'97	0'98	0'99	1'00	1'02
0'8	1'03	1'04	1'05	1'07	1'08	1'09	1'10	1'11	1'13	1'14
0'9	1'15	1'16	1'18	1'19	1'21	1'22	1'23	1'25	1'26	1'28
1'0	1'29	1'31	1'33	1'35	1'36	1'37	1'38	1'40	1'41	1'42

The amount of malt is calculated from the malt extract in the proportion of 32'0 : 21'0, or, if 1,000 be subtracted from the specific gravity, and the remainder multiplied by 0'025, the result will represent the total malt extract in pounds per gallon.

TABLE D.  
SPECIFIC GRAVITY AND PERCENTAGE OF MALT EXTRACT.

Specific Gravity.	Extract per Cent.						
1.0000	0.000	1.0040	1.000	1.0080	2.000	1.0120	3.000
1.0001	0.025	1.0041	1.025	1.0081	2.025	1.0121	3.025
1.0002	0.050	1.0042	1.050	1.0082	2.050	1.0122	3.050
1.0003	0.075	1.0043	1.075	1.0083	2.075	1.0123	3.075
1.0004	0.100	1.0044	1.100	1.0084	2.100	1.0124	3.100
1.0005	0.125	1.0045	1.125	1.0085	2.125	1.0125	3.125
1.0006	0.150	1.0046	1.150	1.0086	2.150	1.0126	3.150
1.0007	0.175	1.0047	1.175	1.0087	2.175	1.0127	3.175
1.0008	0.200	1.0048	1.200	1.0088	2.200	1.0128	3.200
1.0009	0.225	1.0049	1.225	1.0089	2.225	1.0129	3.225
1.0010	0.250	1.0050	1.250	1.0090	2.250	1.0130	3.250
1.0011	0.275	1.0051	1.275	1.0091	2.275	1.0131	3.275
1.0012	0.300	1.0052	1.300	1.0092	2.300	1.0132	3.300
1.0013	0.325	1.0053	1.325	1.0093	2.325	1.0133	3.325
1.0014	0.350	1.0054	1.350	1.0094	2.350	1.0134	3.350
1.0015	0.375	1.0055	1.375	1.0095	2.375	1.0135	3.375
1.0016	0.400	1.0056	1.400	1.0096	2.400	1.0136	3.400
1.0017	0.425	1.0057	1.425	1.0097	2.425	1.0137	3.425
1.0018	0.450	1.0058	1.450	1.0098	2.450	1.0138	3.450
1.0019	0.475	1.0059	1.475	1.0099	2.475	1.0139	3.475
1.0020	0.500	1.0060	1.500	1.0100	2.500	1.0140	3.500
1.0021	0.525	1.0061	1.525	1.0101	2.525	1.0141	3.525
1.0022	0.550	1.0062	1.550	1.0102	2.550	1.0142	3.550
1.0023	0.575	1.0063	1.575	1.0103	2.575	1.0143	3.575
1.0024	0.600	1.0064	1.600	1.0104	2.600	1.0144	3.600
1.0025	0.625	1.0065	1.625	1.0105	2.625	1.0145	3.625
1.0026	0.650	1.0066	1.650	1.0106	2.650	1.0146	3.650
1.0027	0.675	1.0067	1.675	1.0107	2.675	1.0147	3.675
1.0028	0.700	1.0068	1.700	1.0108	2.700	1.0148	3.700
1.0029	0.725	1.0069	1.725	1.0109	2.725	1.0149	3.725
1.0030	0.750	1.0070	1.750	1.0110	2.750	1.0150	3.750
1.0031	0.775	1.0071	1.775	1.0111	2.775	1.0151	3.775
1.0032	0.800	1.0072	1.800	1.0112	2.800	1.0152	3.800
1.0033	0.825	1.0073	1.825	1.0113	2.825	1.0153	3.825
1.0034	0.850	1.0074	1.850	1.0114	2.850	1.0154	3.850
1.0035	0.875	1.0075	1.875	1.0115	2.875	1.0155	3.875
1.0036	0.900	1.0076	1.900	1.0116	2.900	1.0156	3.900
1.0037	0.925	1.0077	1.925	1.0117	2.925	1.0157	3.925
1.0038	0.950	1.0078	1.950	1.0118	2.950	1.0158	3.950
1.0039	0.975	1.0079	1.975	1.0119	2.975	1.0159	3.975

TABLE D (*continued*).

Specific Gravity.	Extract per Cent.	Specific Gravity.	Extract per Cent.	Specific Gravity.	Extract per Cent.	Specific Gravity.	Extract per Cent.
1·0160	4·000	1·0210	5·250	1·0260	6·488	1·0310	7·706
1·0161	4·025	1·0211	5·275	1·0261	6·512	1·0311	7·731
1·0162	4·050	1·0212	5·300	1·0262	6·536	1·0312	7·756
1·0163	4·075	1·0213	5·325	1·0263	6·560	1·0313	7·780
1·0164	4·100	1·0214	5·350	1·0264	6·584	1·0314	7·804
1·0165	4·125	1·0215	5·375	1·0265	6·609	1·0315	7·828
1·0166	4·150	1·0216	5·400	1·0266	6·633	1·0316	7·853
1·0167	4·175	1·0217	5·425	1·0267	6·657	1·0317	7·877
1·0168	4·200	1·0218	5·450	1·0268	6·681	1·0318	7·901
1·0169	4·225	1·0219	5·475	1·0269	6·706	1·0319	7·925
1·0170	4·250	1·0220	5·500	1·0270	6·731	1·0320	7·950
1·0171	4·275	1·0221	5·525	1·0271	6·756	1·0321	7·975
1·0172	4·300	1·0222 <sup>b</sup>	5·550	1·0272	6·780	1·0322	8·000
1·0173	4·325	1·0223	5·575	1·0273	6·804	1·0323	8·024
1·0174	4·350	1·0224	5·600	1·0274	6·828	1·0324	8·048
1·0175	4·375	1·0225	5·625	1·0275	6·853	1·0325	8·073
1·0176	4·400	1·0226	5·650	1·0276	6·877	1·0326	8·097
1·0177	4·425	1·0227	5·675	1·0277	6·901	1·0327	8·122
1·0178	4·450	1·0228	5·700	1·0278	6·925	1·0328	8·146
1·0179	4·475	1·0229	5·725	1·0279	6·950	1·0329	8·170
1·0180	4·500	1·0230	5·750	1·0280	6·975	1·0330	8·195
1·0181	4·525	1·0231	5·775	1·0281	7·000	1·0331	8·219
1·0182	4·550	1·0232	5·800	1·0282	7·024	1·0332	8·244
1·0183	4·575	1·0233	5·825	1·0283	7·048	1·0333	8·260
1·0184	4·600	1·0234	5·850	1·0284	7·073	1·0334	8·292
1·0185	4·625	1·0235	5·875	1·0285	7·097	1·0335	8·316
1·0186	4·650	1·0236	5·900	1·0286	7·122	1·0336	8·341
1·0187	4·675	1·0237	5·925	1·0287	7·146	1·0337	8·365
1·0188	4·700	1·0238	5·950	1·0288	7·170	1·0338	8·389
1·0189	4·725	1·0239	5·975	1·0289	7·195	1·0339	8·413
1·0190	4·750	1·0240	6·000	1·0290	7·219	1·0340	8·438
1·0191	4·775	1·0241	6·024	1·0291	7·244	1·0341	8·463
1·0192	4·800	1·0242	6·048	1·0292	7·268	1·0342	8·488
1·0193	4·825	1·0243	6·073	1·0293	7·292	1·0343	8·512
1·0194	4·850	1·0244	6·097	1·0294	7·316	1·0344	8·536
1·0195	4·875	1·0245	6·122	1·0295	7·341	1·0345	8·560
1·0196	4·900	1·0246	6·146	1·0296	7·365	1·0346	8·584
1·0197	4·925	1·0247	6·170	1·0297	7·389	1·0347	8·609
1·0198	4·950	1·0248	6·195	1·0298	7·413	1·0348	8·633
1·0199	4·975	1·0249	6·219	1·0299	7·438	1·0349	8·657
1·0200	5·000	1·0250	6·244	1·0300	7·463	1·0350	8·681
1·0201	5·025	1·0251	6·268	1·0301	7·488	1·0351	8·706
1·0202	5·050	1·0252	6·292	1·0302	7·512	1·0352	8·731
1·0203	5·075	1·0253	6·316	1·0303	7·536	1·0353	8·756
1·0204	5·100	1·0254	6·341	1·0304	7·560	1·0354	8·780
1·0205	5·125	1·0255	6·365	1·0305	7·584	1·0355	8·804
1·0206	5·150	1·0256	6·389	1·0306	7·609	1·0356	8·828
1·0207	5·175	1·0257	6·413	1·0307	7·633	1·0357	8·853
1·0208	5·200	1·0258	6·438	1·0308	7·657	1·0358	8·877
1·0209	5·225	1·0259	6·463	1·0309	7·681	1·0359	8·901

TABLE D (*continued*).

Specific Gravity.	Extract per Cent.						
1·0360	8·925	1·0410	10·142	1·0460	11·333	1·0510	12·523
1·0361	8·950	1·0411	10·166	1·0461	11·357	1·0511	12·547
1·0362	8·975	1·0412	10·190	1·0462	11·381	1·0512	12·571
1·0363	9·000	1·0413	10·214	1·0463	11·404	1·0513	12·595
1·0364	9·024	1·0414	10·238	1·0464	11·428	1·0514	12·619
1·0365	9·048	1·0415	10·261	1·0465	11·452	1·0515	12·642
1·0366	9·073	1·0416	10·285	1·0466	11·476	1·0516	12·666
1·0367	9·097	1·0417	10·309	1·0467	11·500	1·0517	12·690
1·0368	9·122	1·0418	10·333	1·0468	11·523	1·0518	12·714
1·0369	9·146	1·0419	10·357	1·0469	11·547	1·0519	12·738
1·0370	9·170	1·0420	10·381	1·0470	11·571	1·0520	12·761
1·0371	9·195	1·0421	10·404	1·0471	11·595	1·0521	12·785
1·0372	9·219	1·0422	10·428	1·0472	11·619	1·0522	12·809
1·0373	9·244	1·0423	10·452	1·0473	11·642	1·0523	12·833
1·0374	9·268	1·0424	10·476	1·0474	11·666	1·0524	12·857
1·0375	9·292	1·0425	10·500	1·0475	11·690	1·0525	12·881
1·0376	9·316	1·0426	10·523	1·0476	11·714	1·0526	12·904
1·0377	9·341	1·0427	10·547	1·0477	11·738	1·0527	12·928
1·0378	9·365	1·0428	10·571	1·0478	11·761	1·0528	12·952
1·0379	9·389	1·0429	10·595	1·0479	11·785	1·0529	12·976
1·0380	9·413	1·0430	10·619	1·0480	11·809	1·0530	13·000
1·0381	9·438	1·0431	10·642	1·0481	11·833	1·0531	13·023
1·0382	9·463	1·0432	10·666	1·0482	11·857	1·0532	13·047
1·0383	9·488	1·0433	10·690	1·0483	11·881	1·0533	13·071
1·0384	9·512	1·0434	10·714	1·0484	11·904	1·0534	13·095
1·0385	9·536	1·0435	10·738	1·0485	11·928	1·0535	13·119
1·0386	9·560	1·0436	10·761	1·0486	11·952	1·0536	13·142
1·0387	9·584	1·0437	10·785	1·0487	11·986	1·0537	13·166
1·0388	9·609	1·0438	10·809	1·0488	12·000	1·0538	13·190
1·0389	9·633	1·0439	10·833	1·0489	12·023	1·0539	13·214
1·0390	9·657	1·0440	10·857	1·0490	12·047	1·0540	13·238
1·0391	9·681	1·0441	10·881	1·0491	12·071	1·0541	13·261
1·0392	9·706	1·0442	10·904	1·0492	12·095	1·0542	13·285
1·0393	9·731	1·0443	10·928	1·0493	12·119	1·0543	13·309
1·0394	9·756	1·0444	10·952	1·0494	12·142	1·0544	13·333
1·0395	9·780	1·0445	10·976	1·0495	12·166	1·0545	13·357
1·0396	9·804	1·0446	11·000	1·0496	12·190	1·0546	13·381
1·0397	9·828	1·0447	11·023	1·0497	12·214	1·0547	13·405
1·0398	9·853	1·0448	11·047	1·0498	12·238	1·0548	13·428
1·0399	9·877	1·0449	11·071	1·0499	12·261	1·0549	13·452
1·0400	9·901	1·0450	11·095	1·0500	12·285	1·0550	13·476
1·0401	9·925	1·0451	11·119	1·0501	12·309	1·0551	13·500
1·0402	9·950	1·0452	11·142	1·0502	12·333	1·0552	13·523
1·0403	9·975	1·0453	11·166	1·0503	12·357	1·0553	13·547
1·0404	10·000	1·0454	11·190	1·0504	12·381	1·0554	13·571
1·0405	10·023	1·0455	11·214	1·0505	12·404	1·0555	13·595
1·0406	10·047	1·0456	11·238	1·0506	12·428	1·0556	13·619
1·0407	10·071	1·0457	11·261	1·0507	12·452	1·0557	13·642
1·0408	10·095	1·0458	11·285	1·0508	12·476	1·0558	13·666
1·0409	10·119	1·0459	11·309	1·0509	12·500	1·0559	13·690

TABLE D (*continued*).

Specific Gravity.	Extract per Cent.						
1·0560	13·714	1·0595	14·547	1·0630	15·371	1·0665	16·186
1·0561	13·738	1·0596	14·571	1·0631	15·395	1·0666	16·209
1·0562	13·761	1·0597	14·595	1·0632	15·418	1·0667	16·232
1·0563	13·785	1·0598	14·619	1·0633	15·441	1·0668	16·255
1·0564	13·809	1·0599	14·642	1·0634	15·464	1·0669	16·278
1·0565	13·833			1·0635	15·488		
1·0566	13·857	1·0600	14·666	1·0636	15·511	1·0670	16·302
1·0567	13·881	1·0601	14·690	1·0637	15·534	1·0671	16·325
1·0568	13·904	1·0602	14·714	1·0638	15·557	1·0672	16·348
1·0569	13·928	1·0603	14·738	1·0639	15·581	1·0673	16·371
		1·0604	14·761	—	—	1·0674	16·395
1·0570	13·952	1·0605	14·785	1·0640	15·604	1·0675	16·418
1·0571	13·976	1·0606	14·809	1·0641	15·627	1·0676	16·441
1·0572	14·000	1·0607	14·833	1·0642	15·650	1·0677	16·464
1·0573	14·023	1·0608	14·857	1·0643	15·674	1·0678	16·488
1·0574	14·047	1·0609	14·881	1·0644	15·697	1·0679	16·511
1·0575	14·071			1·0645	15·721		
1·0576	14·095	1·0610	14·904	1·0646	15·744	1·0680	16·534
1·0577	14·119	1·0611	14·928	1·0647	15·767	1·0681	16·557
1·0578	14·142	1·0612	14·952	1·0648	15·790	1·0682	16·581
1·0579	14·166	1·0613	14·976	1·0649	15·814	1·0683	16·604
		1·0614	15·000	—	—	1·0684	16·627
1·0580	14·190	1·0615	15·023	1·0650	15·837	1·0685	16·650
1·0581	14·214	1·0616	15·046	1·0651	15·860	1·0686	16·674
1·0582	14·238	1·0617	15·070	1·0652	15·883	1·0687	16·697
1·0583	14·261	1·0618	15·093	1·0653	15·907	1·0688	16·721
1·0584	14·285	1·0619	15·116	1·0654	15·930	1·0689	16·744
1·0585	14·309			1·0655	15·953		
1·0586	14·333	1·0620	15·139	1·0656	15·976	1·0690	16·767
1·0587	14·357	1·0621	15·162	1·0657	16·000	1·0691	16·790
1·0588	14·381	1·0622	15·186	1·0658	16·023	1·0692	16·814
1·0589	14·404	1·0623	15·209	1·0659	16·046	1·0693	16·837
		1·0624	15·232	—	—	1·0694	16·860
1·0590	14·428	1·0625	15·255	1·0660	16·070	1·0695	16·883
1·0591	14·452	1·0626	15·278	1·0661	16·093	1·0696	16·907
1·0592	14·476	1·0627	15·302	1·0662	16·116	1·0697	16·930
1·0593	14·500	1·0628	15·325	1·0663	16·139	1·0698	16·953
1·0594	14·523	1·0629	15·348	1·0664	16·162	1·0699	16·976
				—	—	1·0700	17·000

## EXAMPLES.

I.	Specific gravity of alcoholic distillate	..	..	..	1000°
					993·9
Spirit indication	..	..	..	..	6·1
The spirit indication 6·1 is equal to 24·6 (Table A).					

Gravity of boiled beer	..	..	..	..	..	1014·7
Add	..	..	..	..	..	24·6
						1038·3

Original gravity 1038·3 is equal to 9·488 per cent. malt extract (Table D).

2. By the evaporation method a beer was found to contain 5·94 per cent. alcohol = specific gravity, 0·9899 (see Alcohol Tables in the Appendix).

						1000·0
						989·9
Spirit indication	..	..	..	..	..	10·1

The spirit indication 10·1 is equal to 44·7 (Table A).

Gravity of boiled beer	..	..	..	..	..	1012·3
Add	..	..	..	..	..	44·7
Original gravity	..	..	..	..	..	1057·0

Original gravity 1057·0 is equal to 13·952 per cent. malt extract (Table D).

3. The loss of gravity by acetic acid is ascertained by reference to Table C. Thus, 0·48 per cent. acetic acid was found in Example I. From this deduct 0·10 per cent., an amount which is usually allowed for as being naturally formed in normal fermentation, and allowed for in the table. Then—

Total acetic acid	..	..	..	..	..	0·48
Less	..	..	..	..	..	0·1
Acetic acid to be accounted for	..	..	..	..	..	0·38

0·38 is equal to 0·49 by Table C, so that this number must be added to the spirit indication.

Spirit indication from alcohol	..	..	..	..	..	6·1
Spirit indication from acetic acid	..	..	..	..	..	0·38
Total spirit indication	..	..	..	..	..	6·48

Finish the calculation as in Example No. I.

**Hop-Resin and Glycerine.**—Concentrate the beer by evaporation to about one-third its original volume, and extract the hop-resin by shaking up with petroleum ether in a separating-funnel.

Pour off the ether into a tared dish, evaporate, and weigh the residual resin. Make the resin-free residue alkaline with barium hydroxide, and extract the glycerine by shaking with a mixture of 3 parts of ether and 2 parts of alcohol.

**The Bitter used.**—This is necessarily a tedious operation, and for full particulars the reader is referred to such books upon poisons as describe the processes of Dragendorff and others.

The prepared and concentrated beer is subjected to a series of extractions with petroleum ether, benzene, chloroform, and amyl alcohol, each of which is examined in turn.

## CHAPTER XIV

### EXAMINATION AND ANALYSIS OF WATER

**Sampling.**—All samples should be taken in glass bottles, preferably those known as "Winchester quarts"; stoneware jars are to be avoided. The "Winchesters" should have well-ground stoppers; if corked, the corks must be new, and have been previously well rinsed in pure water. One Winchester quart is usually sufficient for an ordinary analysis suitable for dietetic purposes, but when the Frankland process is used two bottles, at least, must be taken.

In sampling from a river or tank, the bottle is immersed and rinsed out several times, care being taken to avoid the introduction of any mud or sediment.

In sampling from a pipe, allow the water to flow for several minutes at full bore, and then, after rinsing out the sample bottle, fill it to the top, insert the stopper, and tie it down with a piece of clean muslin.

Note the condition of the stream or other source at the time of sampling.

**Preliminary Observations.**—The colour is observed in a 2-foot clear glass tube standing upon a white plate or in a colorimeter.

The appearance of the water is carefully noted, especially whether it is turbid or clear and bright.

The turbidity is best observed by filling a clean litre flask with the sample and holding it up to the light. The purest waters possess a bluish tint; next to these are waters which exhibit a greenish tinge. Peaty waters are tinted brown, owing to the presence of finely divided organic matter.

An ingenious colorimeter is that invented by Messrs. Crookes and Odling, consisting of two hollow wedges filled respectively

with brown and blue solutions. By superimposing these wedges any desired tint may be matched. The prisms are graduated 1 to 50, representing the thickness in millimetres of the prisms at any given point.

A small portion of the sample is slowly warmed to 38° C., and the odour observed.

It is advisable always to taste water samples, although only a few have decided tastes.

The action upon litmus is noted.

**Estimation of the Suspended Matter.**—This is not necessary unless the water is turbid. Steep a filter-paper (6-inch) in water free from ammonia for about a day, and filter 1 to 2 litres of the sample, rejecting the first portion of the filtrate. In cases where the water is much polluted or in sewage the ammonia in the filter may be neglected, as it is very small. Dry the filter and contents in the air-oven at 100° C. until the weight is constant.

Next cut up the dried filter, and gently ignite it in a tared platinum dish until the residue contains no organic matter, cool, and weigh. The latter weight represents the amount of inorganic suspended matter in the amount taken. Calculate this into parts per 100,000.

**Microscopical Appearance.**—When there is any suspended matter present it is often advisable to make a microscopic examination.

A convenient method is to fill a tall cylinder with the sample, and allow the suspended matter to settle during the ensuing twenty-four hours. Carefully decant off the clear liquid, and transfer the sediment to a slip for examination.

**Total Solids.**—If the water has been filtered, a portion of the clear filtered liquid is operated upon. The amount depends entirely upon the nature of the sample. With calcareous waters 100 c.c. is amply sufficient; with soft waters—*e.g.*, Loch Katrine—500 c.c. is necessary. It is advisable in dealing with an unknown sample to take 250 c.c. This is placed in a tared platinum evaporating-basin and gently heated, care being taken to prevent boiling. The last drops are expelled on a water-bath. The basin and contents are then dried in an air-oven at a temperature of 104° C. until the weight is constant. The solids are next heated at a dull red heat to expel any organic matter, and the loss in weight thus in-

curred is reported as "loss on ignition." The inorganic constituents thus remaining can be further examined for the presence of phosphates, iron, etc.

**Estimation of Chlorine.**—The amount of chlorine in water is usually very small, unless in the vicinity of salt-beds, the sea, or sewage contamination.

Place 50 c.c. of the sample in a clear porcelain evaporating-basin, add 2 to 3 drops of a solution of potassium chromate free from chlorine, and titrate with a standard solution of silver nitrate, prepared by dissolving 2·3944 grammes pure recrystallized silver nitrate in distilled water, and making the solution up to 1 litre. Each c.c. of this solution represents 1 part of chlorine per 100,000, operating on 50 c.c. of the water sample.

Should the number of c.c. used exceed 10, it is advisable to work upon a smaller quantity of the sample.

The exact end-point is best observed through a cell containing a weak chromate solution, but with a little practice even this is unnecessary.

**Estimation of Nitrates and Nitrites—I. As Ammonia by the Zinc-Copper Couple.**—Pour a 3 per cent. solution of crystallized copper sulphate upon a piece of clean zinc-foil 3 inches by 2 inches, and allow the deposition to proceed until a heavy, firm black layer of copper is formed. Wash the prepared copper thoroughly, and finally allow it to drain.

Place the couple in a 10-ounce stoppered fractionating-flask, and connect the tubule of the flask with a U-tube filled with pumice moistened with hydrochloric acid, both the latter having been previously freed from ammonia.

A measured quantity of the water is now added to the flask, and allowed to stand for one hour.

Messrs. Gladstone and Tribe have shown that the action of the zinc-copper couple upon the nitrate solution consists in its electrolysis, resulting in the evolution of hydrogen, whereby the nitrate is first reduced to nitrite and then into ammonia.

It is necessary, therefore, to test the solution for the presence of any undecomposed nitrite by mixing a small portion with dilute sulphuric acid and metaphenylenediamine; then add about 1 gramme of freshly ignited sodic carbonate, and wash in the

pumice containing the acid from the U-tube. Now connect the flask with a Liebig condenser, and proceed to estimate the ammonia by the Wanklyn process.

2. As *Nitric Oxide* (*Crum's Process*).—Five hundred c.c. of the water are evaporated to dryness over the water-bath, and the residue



FIG. 36.—ESTIMATION OF NITROGEN BY NITROMETER.

extracted with hot water. The aqueous extract is concentrated to 1 c.c., and transferred to a nitrometer. The nitrometer having previously been filled with mercury, the extract is washed into the cup with as little water as possible.

Allow the solution to pass through the stopcock, allowing no air to pass with it. Introduce in this way double its volume of pure

strong sulphuric acid free from nitrogen. Allow both liquids to mix ; heat is evolved, and bubbles of carbon dioxide rise to the surface. As soon as these cease, raise the pressure-tube and expel all the gas, immediately shutting the stopcock.

Next agitate the contents of the nitrometer in such a way that the acid liquid is intimately mixed with the mercury for a period of at least ten minutes, with constant shaking. Allow to stand until all evolution of gas ceases, and the liquid has cooled down to the temperature of the laboratory.

Adjust the levels of the mercury in both columns so that the pressure is exactly atmospheric. Read off the volume of the gas, and note the existing temperature and pressure.

Every two volumes of nitric oxide are equal to one volume of nitrogen. The weight of the nitrogen is obtained in this way by reference to the table appended, or the volume of the gas obtained in c.c., corrected to normal temperature and pressure, when multiplied by the factor 0.175, gives the nitrogen in nitrates in grains per gallon, using 250 c.c. of the sample.

3. *The Indigo Process.*—This is the most convenient process, giving results which with care compare very favourably with the Crum process.

(a) Standard potassic nitrate of  $\frac{N}{1000}$  strength, prepared by dissolving 0.1011 gramme  $KNO_3$  in 1 litre pure distilled water.

(b) Indigo carmine, preferably Gehe's (sodie sulphindylate), which will not give a brown colour with nitric acid. Dissolve 1 gramme in 500 c.c. pure dilute sulphuric acid (1 : 20). Store this solution in the dark.

(c) Pure concentrated sulphuric acid free from nitrogen.

*Process.*—Place 10 c.c. of the standard nitrate in a 150-c.c. flask, add 10 e.e. indigo, and rapidly thereafter 20 e.e. of the sulphuric acid. Mix, and quickly heat over a Bunsen burner. If the solution is decolorized, more indigo is added, with constant stirring, until after heating for about half a minute a persistent greenish tint occurs. The number of e.e.'s of indigo is noted, and is repeated until an accurate estimate of the degree of dilution of the indigo solution is ascertained. This standardization is repeated each day the indigo is used, and the "blank" thus obtained is set aside as a standard comparison for the "greenish shade."

Next place 10 c.c. of the water sample in a similar flask, add a known quantity of the indigo from a burette, and 20 c.c. of the sulphuric acid. Heat as before; add more indigo if necessary. If overdone, repeat the trial with less indigo until the exact amount of indigo solution is obtained by comparison test with the "blank" already mentioned. It is important that the length of time taken in heating—say thirty seconds—should always be carefully marked and adhered to.

Oxidized indigo is of a clear yellow colour.

**Estimation of Nitrite.**—Prepare the following solutions :

1. *Metaphenylenediamine.*—Dissolve 5 grammes in 1,000 c.c. distilled water which has been acidified with a few drops of pure sulphuric acid.

2. *Dilute Sulphuric Acid.*—Mix 1 part concentrated sulphuric acid with 2 parts distilled water.

3. *Sodium Nitrite.*—Dissolve 0·406 gramme pure silver nitrite in boiling distilled water, add excess pure sodic chloride solution, make up to 1,000 c.c. with distilled water, allow to settle, and make up 100 c.c. of this clear solution to 1,000 c.c. with distilled water. Alternatively, dissolve 0·1823 gramme pure sodic nitrite in 1,000 c.c. distilled water. Dilute 100 c.c. of this solution to 1,000 c.c., when 1 c.c. of this dilute solution will equal 0·00001 gramme  $N_2O_3$ . Store solutions in a dark place.

*Process.*—Measure 100 c.c. of the sample into a colourless glass cylinder capable of holding 150 c.c., add 1 c.c. dilute acid, and then 1 c.c. S.S. metaphenylenediamine, and stir well. Compare the tint thus produced with a similar cylinder containing distilled water to which the required amount of S.S. nitrite has been added from a burette after the lapse of twenty minutes. As the colour alters in depth, a second trial is advisable under the same conditions and simultaneously. Calculate to parts per 100,000.

**Detection of Nitrates and Nitrates in Water.**—The following is a very useful test in cases where the presence of nitrates and nitrates is uncertain or of not enough consequence to justify a quantitative test :

Take distilled water, a water of known freedom from nitrates and nitrates, or at least practically so, and the sample in three separate conical test-glasses of  $2\frac{1}{2}$  inches diameter. Add to each a pinch of

pyrogallop, slope the glass, and pour down slowly pure sulphuric acid free from nitrogen (about 5 c.c.); then add a pinch of pure sodic chloride, and observe the reaction against a white background.

Any evolution of red fumes indicates the presence of nitrites; a red coloration in the vitriol shows nitrates.

**Estimation of Oxygen consumed by Organic Matter (Forschammer Process).**—Prepare the following solutions:

1. *Potassium Permanganate*.—Dissolve 0.395 grammes pure  $\text{KMnO}_4$  in 1,000 c.c. distilled water. Each c.c. of this solution contains 0.001 grammes available oxygen.

2. *Sodium Thiosulphate*.—Dissolve 1 grainme in 1,000 c.c. distilled water.

3. *Dilute Sulphuric Acid*.—Mix 1 part pure acid with 3 parts distilled water.

4. *Potassic Iodide*.—Dissolve 1 part in 10 parts distilled water.

5. *Starch*.—A clear, thin, cold aqueous solution (1 : 100).

*Process*.—Take two thoroughly clean 18-ounce Erlenmeyer flasks. Place in one 250 c.c. pure distilled water, and in the other 250 c.c. of the sample. Add to each 10 c.c. S.S.  $\text{KMnO}_4$  and 10 c.c. of the dilute sulphuric acid solution. Cover with watch-glasses, and allow to stand for three hours in a dark place. If the pink colour disappears during this interval the process must be repeated, using 20 c.c. or more of the S.S.  $\text{KMnO}_4$ . Then add to the first flask a little of the iodide solution, and titrate with the thiosulphate until the liquid assumes a pale straw tint; add a few drops of the starch solution and more thiosulphate, drop by drop, until the blue colour just disappears.

Treat the sample flask in the same manner.

*Calculation*.—Let  $x$  equal the number of c.c. S.S. thiosulphate used in titrating the distilled water, also  $y$  the number of thiosulphate in the case of the sample; then  $x - y$  will equal the number of c.c. of thiosulphate equivalent to the permanganate reduced by the organic matter. Supposing that 10 c.c. of the S.S.  $\text{KMnO}_4$  were used, and representing this by the symbol  $z$ , then the weight of oxygen required to oxidize the organic matter in the 250 c.c. of the sample will be  $(x - y)z$ .

But  $z$  equals 0.001 gramme oxygen; therefore the quantity of oxygen consumed by 100,000 parts of the sample is—

$$\frac{(x-y)z \times 0.001 \times 100,000}{x \times 250} = \frac{(x-y) 0.4}{x}.$$

**Estimation of Hardness.**—"Hardness" of water, or soap-dissolving power, is due to the presence of dissolved salts of lime and magnesia. These combine with the fatty acids, forming insoluble curd. Other impurities also prevent the formation of lather—e.g., chlorides, which precipitate the soap unaltered.

Hardness is of two kinds—"temporary hardness," which can be removed by boiling, and is due to the presence of soluble bicarbonates; and "permanent hardness," which is not removed by boiling, but can be got rid of by treatment with alkalies.

The hardness in water may be estimated by two methods—first, Clark's process; secondly, Hehner's process.

**Clark's Process.**—This is the older process, and does not give accurate results when excess of magnesium salts are present, owing to the fact that magnesium salts require more soap solution than lime in a given time, and form a scum on the surface of the solution.

Prepare the following solutions :

1. *Calcic Chloride.*—Dissolve 0.2 gramme pure Iceland spar in dilute hydrochloric acid, and evaporate the solution to dryness on the water-bath, covering the basin with a clock-glass to avoid spitting. Take up in water and again evaporate the solution to dryness, and repeat this process three times. Finally, take up with hot distilled water, and make the solution up to 1,000 c.c. at 15.5° C.

2. *Soap Solution.*—Dissolve 10 grammes good Castile-soap shavings in a litre of methylated spirit or 35 per cent. alcohol. Dilute this solution until 14.25 c.c. just cause a permanent lather with 50 c.c. S.S. calcic chloride in this way. Measure 50 c.c. of the standard hard water into a 250-c.c. wide-mouth stoppered bottle (the stopper must be accurately ground in, and the bottle reserved for this purpose only); allow the solution to flow in from the pipette by gravitation, else blowing from the mouth will cause a precipitation of the calcium carbonate.

Then add the soap solution gradually, about 1 c.c. at a time,

vigorously shaking the bottle after each addition. Fresh additions are made until a lather begins to appear, which persists when the bottle is placed on its side. At this stage the soap must be added cautiously drop by drop, until the lather is persistent, and no bubbles are seen to break on the surface when the bottle is laid at rest on its side for a period of two minutes.

The soap solution is then finally adjusted, so that 14·25 c.c. produce a "permanent" lather in this way with 50 c.c. of the standard calcic chloride solution.

**Estimation of Total Hardness—Process.**—Place 50 c.c. of the sample under consideration in the test-bottle already described, after it has been rinsed free from the last operation with the aid of a little distilled water, and proceed by adding the soap solution gradually, and vigorously shaking after each addition, as already explained.

If more than 16 c.c. of the soap solution are required, a suitable quantity must be taken of the sample, and diluted to 50 c.c., with the addition of distilled water. This is necessary, because when the amount exceeds 16 c.c. S.S. soap the end-point is indistinct, and the total volume of water taken must always be 50 c.c., whether diluted or otherwise.

The degree of hardness (total) in the sample is thus obtained by reference to the hardness table (appended).

**Estimation of the Permanent Hardness.**—Measure 250 c.c. of the sample into an Erlenmeyer flask, and boil it gently for thirty minutes, adjusting the amount from time to time by the addition of freshly boiled distilled water. Cool the contents of the flask as quickly as possible, make up to the original volume with cold freshly boiled distilled water, mix, and filter through a dry filter into a dry vessel.

Titrate 50 c.c. as already described.

The number of degrees thus obtained by reference to the hardness table represents the permanent hardness in the sample.

**Temporary Hardness.**—The difference between the degree of total hardness and permanent hardness yields the temporary hardness of the sample.

**Hehner's Process for Hardness.**—Prepare the following solutions :

1. *Sulphuric Acid.*—Twenty c.c.  $H_2SO_4$  made up to 1,000 c.c. with distilled water.

2. *Sodic Carbonate.*—A solution (1:06 : 1,000) exactly equal to the above sulphuric acid in strength.

One c.c. of the acid is equal to 10 milligrammes calcic chloride.

*Process.*—Titrate 100 c.c. of the sample with the acid, using as the indicator methyl orange. Each c.c. used indicates 1 degree of temporary hardness.

To another 100 c.c. of the sample a known amount of the sodic carbonate is added, more than sufficient to decompose all the soluble lime and magnesian salts. The resulting solution is evaporated to dryness in a platinum basin, extracted with hot water, filtered, and titrated whilst still warm with the S.S. sulphuric acid. The alkali added, minus the acid used, gives the permanent hardness expressed as calcic carbonate.

**Estimation of Ammonia.**—The nitrogen present in water may be in one or both of two forms, known as, first, "free ammonia," and, second, "albuminoid ammonia"—free ammonia existing either as ammonia or ammoniacal salts (saline), and albuminoid ammonia, that, in combination with nitrogenous organic matter.

The process consists essentially in distilling the ammonia in the presence of aqueous vapour from a very slightly alkaline solution, and testing the distillate with a sensitive indicator—viz., Nessler's solution.

For this purpose it is necessary, therefore, to have a large (preferably two) well-fitting stoppered glass retort connected with a long Liebig's condenser through which a strong current of water passes constantly. Also a series of measuring-flasks and pipettes, and one burette retained for this process only; several 50-c.c. and 100-c.c. "Nessler glasses," all of the same diameter and thickness (this is very important for comparing equal volumes); and, finally, the following solutions :

\*1. *Nessler's Solution.*—Dissolve 62.5 grammes potassium iodide in about 250 c.c. distilled water. Set aside a few c.c. of this solution, add with constant stirring to the main solution a cold saturated solution of mercuric chloride until a permanent precipitate appears. Carefully redissolve this precipitate (with the exception of a faint trace) by adding the reserved portion of the iodide solution drop

by drop with constant stirring. Add 150 grammes potassium hydrate previously dissolved in 150 c.c. distilled water in the cold, and allow the Nessler solution thus prepared to stand over for some days in the dark in a well-stoppered bottle. Decant (do not use a pipette) a portion for use, after settling, into a reagent bottle provided with a rubber cork through which a glass tube passes under the surface of the solution, and marked 1 c.c. and 2 c.c. Fill this pipette by pushing down into the solution, closing the

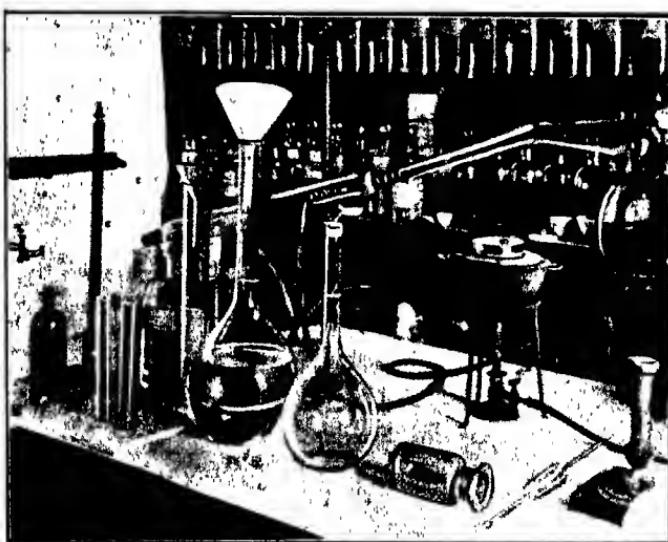


FIG. 37.—WATER ANALYSIS (WANKLYN PROCESS).

open end with the finger when filled to the required mark, and transferring the quantity to the test-glass. Do not suck the solution up into the pipette.

**2. Standard Ammonium Chloride Solution.**—Dissolve 1.5735 grammes pure resublimed ammonium chloride in 1,000 c.c. distilled water. This forms the "stock solution." Dilute this solution as may be required by taking 100 c.c. and making it up to 1,000 c.c. with distilled water (free from ammonia).

Each c.c. of the working solution (diluted solution) contains 0.00005 grammes NH<sub>3</sub>.

3. *Ammonia-free Water.*—Fill a large clean stoppered retort with fresh water and distil, collecting the distillates in 50-c.c. Nessler glasses ; as each glass is filled add 2 c.c. Nessler solution, and place the glass on a clean white tile. When the distillate shows no brown coloration, proceed to collect all the water that comes over subsequently, and store it in a well-stoppered bottle in a cupboard away from any trace of ammonia fumes as "ammonia-free water."

4. *Alkaline Potassium Permanganate Solution.*—Dissolve 10 grammes KOH and 5 grammes KMnO<sub>4</sub> in 50 c.c. distilled water in a flask, and boil until the volume is reduced by one-half. Carry this out while the free ammonia is being estimated.

A larger quantity may be prepared and freed from ammonia as may be required.

**Estimation of the Free Ammonia.**—Fifty c.c. of the sample are first tested with 2 c.c. of the Nessler reagent ; if it shows any decided colour it may be necessary to distil a very small portion—say 250 c.c.—of the sample. If there is no appreciable tint, 1,000 c.c. of the sample should be taken and placed in the retort, together with a pinch of freshly ignited sodic carbonate. The retort must have been previously freed from ammonia by boiling and testing the distillates till free with Nessler solution. Having charged the retort with the sample, boil its contents briskly over a rose flame or Argand burner, and collect each 50 c.c. of the distillate in the Nessler glasses, taking care, however, that all aqueous vapour is condensed. The glasses should be rinsed out repeatedly and previously to use, and not under any circumstances wiped inside. Each distillate is tested in turn by adding 2 c.c. Nessler solution, and when no coloration is found the distillation is stopped, as all the free ammonia has then passed over.

In a pure water this usually happens after the first, the purest waters containing no free ammonia. It sometimes happens, notably in the case of sewage, that the later distillates contain more ammonia than the first. In any case, all the free ammonia must be distilled over.

Now pour into a clean Nessler glass 50 c.c. ammonia-free water, and add as much S.S. ammonium chloride from the burette as will

produce the same depth of brown tint when treated with 2 c.c. Nessler solution. It is imperative that the ammonia be added to the water before the Nessler solution, otherwise a turbidity will be produced ; nor must more ammonia solution be added to the glass after the Nessler solution has been already introduced. If the shades do not correspond, the comparison must be repeated with a fresh quantity of ammonia-free water, adding more or less S.S. ammonia as may be required. After a little practice it is quite easy to guess almost, if not altogether, to the tenth of a c.c. how much S.S. ammonium chloride is required to produce any given brown coloration.

The total number of c.c. of ammonia thus required in all the distillates added together represents the amount of free ammonia present in the sample. Thus—

First cylinder required 0·7 c.c. S.S.  $\text{NH}_4\text{Cl}$ .

Second cylinder required 0·2 c.c. S.S.  $\text{NH}_4\text{Cl}$ .

\*Third cylinder required nil c.c. S.S.  $\text{NH}_4\text{Cl}$ , showing no brown tint with Nessler.

$$\begin{aligned}\therefore \text{the } 500 \text{ c.c. taken of the sample} &= 0·9 \text{ c.c. S.S. } \text{NH}_4\text{Cl} \\ &= 0·9 \times 0·00005 \text{ grammes } \text{NH}_3 \\ &= 0·000045 \text{ grammes } \text{NH}_3.\end{aligned}$$

And  $0·000045 \times 200 = 0·009$  part  $\text{NH}_3$  per 100,000.

**Estimation of the Albuminoid Ammonia.**—To the residue remaining in the retort the alkaline potassium permanganate solution already described, and freed from ammonia, is rapidly added, together with a coil of clean recently heated platinum wire, to prevent bumping, and the process proceeded with as before. An alternative method is to place 500 c.c. of distilled water in a clean retort, and together with the required amount of permanganate solution, to distil until the distillate shows no trace of ammonia when tested with Nessler solution. This latter method I prefer, and then add a measured amount, usually half that taken for the free ammonia estimation, and proceed to distil as before.

In the first instance the ammonia obtained will be the albuminoid ammonia only, and in the latter that of the total ammonia, from which the albuminoid ammonia may be ascertained by subtracting from the total the free already found.

Calculate the results as before into parts per 100,000.

**Estimation of Organic Carbon and Nitrogen (Frankland's Combustion Process).**—This is estimated immediately after the Wanklyn (ammonia) process.

If the amount of nitrogen is less than 0·05 part per 100,000, a litre is evaporated; if more than 0·05, and less than 0·2, half a litre; if more than 0·2, and less than 1·0, then 100 c.c. of the sample is taken, or even less.

Measure out the requisite amount of the sample into a Bischof evaporating apparatus, together with 15 c.c. sulphurous acid.

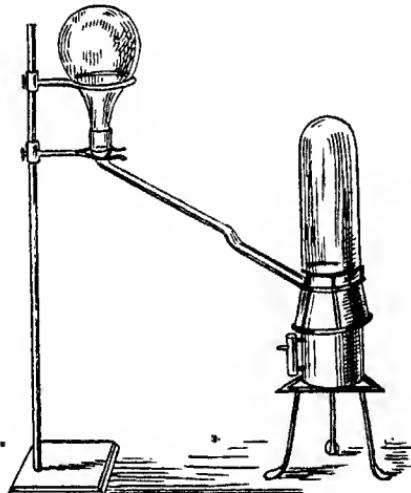


FIG. 38.—BISCHOF EVAPORATING APPARATUS.

In the case of sewage it is better to use hydric metaphosphate in place of sulphurous acid, as the ammonic phosphate is less volatile than the sulphite in cases where nitrates and nitrites are absent.

With a constant-level water-bath operating upon 1,000 c.c. the evaporation may be accomplished overnight.

Prepare a number of Jena glass combustion-tubes having an internal diameter of 10 millimetres and length about 450 millimetres by rinsing them out with water and then alcohol several times. Detach the residue from the glass evaporating-basin with the aid

of a flexible steel spatula, and thoroughly mix it with ground cupric oxide. Transfer it to a combustion-tube.

It is preferable to prepare a series of combustion-tubes at one time, and to store them in a tightly fitting case away from dust.

The residue plus cupric oxide should occupy about 30 millimetres of the length of the tube. Rinse out the dish with a little more oxide, and fill up the tube to about half its capacity with coarse granulated oxide, or, better still, finely cut cupric oxide wire. Insert a small coil of copper gauze, and finally a little more of the coarse granules or wire. Now draw out the open end for a distance of about 100 millimetres, having a bore of about 4 millimetres, and bend it at right angles thus :

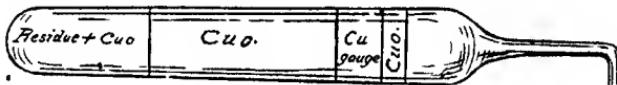


FIG. 39.—COMBUSTION-TUBE.

Round off the edges. This tube can be coupled to the Sprengel pump by means of thick-walled indiarubber tubing and a little vaseline or glycerine in such a way as to make a tight joint.

The tube having been placed *in situ* in the combustion-furnace, and coupled on to the mercury pump in the manner mentioned, a vacuum is set up in the apparatus. This is ascertained when the mercury falls with a loud clicking sound. As soon as this is attained the pump is stopped, and a test-tube filled with mercury is placed over the delivery-tube of the pump. The combustion is now proceeded with in the usual way by slowly turning on the burners one after another, and finally heating the whole tube to redness for a period of about twenty minutes, the combustion occupying from fifty to sixty minutes.

While the "whole" tube is being heated the gases may be drawn off and transferred to the gas analysis apparatus after complete exhaustion.

The gases consist of carbonic anhydride, nitric oxide, nitrogen, and occasionally carbonic oxide. These are now estimated in the Frankland gas apparatus. This consists in a measuring-tube which is accurately calibrated, and stands, together with the pressure-tube, in a column of water. The "laboratory vessel" is connected with this measuring-tube by means of a tightly fitting steel joint,

and both it and the measuring-tube are provided with accurately ground stopcocks. Joints and cocks should be slightly smeared with resin cerate.

Preparatory to the introduction of the gases, the laboratory vessel having been filled with mercury and connected with the measuring-tube, the reservoir is raised in order to expel all bubbles of air. The reservoir is next lowered, and the stopcocks are closed, when the gases are introduced by upward displacement from the test-tube into the laboratory vessel. The cocks are then opened,

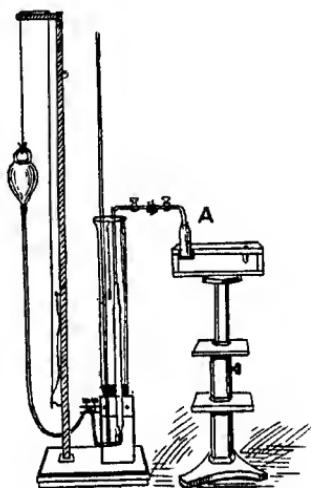


FIG. 40.—FRANKLAND GAS APPARATUS.

and the gases drawn over into the measuring-tube and measured, the temperature being carefully noted to  $\frac{1}{10}$ ° C. at each measurement. The atmospheric pressure for the day is also noted.

It may be that the gases contain a little sulphurous anhydride. This is best observed and estimated, if need be, by returning the gases to the laboratory vessel and adding a drop or two of the potassic bichromate solution by means of the pipette provided with a bent delivery-tube.

If sulphurous anhydride be present, the yellow solution will turn green in colour, and the gases may be again measured in order to

ascertain the true volume and loss due to the absorption of the sulphurous anhydride.

The absorption of the carbonic anhydride is now carried out by the introduction into the laboratory vessel of a few drops of the saturated potassic hydrate solution by means of the pipette. Care must be taken that none of the absorbent solutions are allowed over into the measuring-tube. They must in no case extend beyond the stopcock of the measuring-tube, from which point the gas is measured. As the carbonic anhydride absorption is somewhat rapid, at the end of about five minutes the residual gases, which consist of nitric oxide and nitrogen, may be measured.

Return these two gases after measurement to the laboratory vessel, and introduce a saturated solution of pyrogallic acid. If oxygen be present the pyrogallol will be darkened; in this case introduce a few bubbles of pure oxygen to oxidize the nitric oxide

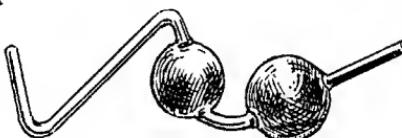


FIG. 41.—GAS PIPETTE.

into pernitric oxide, which will in the latter condition be absorbed by the alkali.

The residual nitrogen after cooling is finally measured.

From these experiments we ascertain—

- (a) Volume of the three mixed gases.
- (b) Volume of nitric oxide and nitrogen.
- (c) Volume of nitrogen.

These volumes must be reduced to  $0^{\circ}$  C. and 760 millimetres pressure, and from these corrected volumes of nitrogen and carbonic oxide the weights of nitrogen and carbon are calculated:

If  $a$  = weight of total gas,  
 $b$  = weight of nitric oxide and nitrogen,  
 $c$  = weight of nitrogen,  
then the weight of carbon will be  $(a - b)^{\frac{2}{3}}$ , and the weight  
of nitrogen will be  $\frac{b - c}{2}$ .

Again, 1 c.c. nitrogen weighs at  $0^{\circ}$  C. and 760 millimetres pressure 0.0012562 gramme, and the formula, therefore, is—

$$W = \frac{0.0012562 \times v \times p}{(1 + 0.00367 t) 760}$$

where W is the weight of nitrogen, v the volume, p the pressure corrected for tension of aqueous vapour, and t the temperature in degrees C.

The following is an example of an analysis of a sample of Chelsea water calculated by the aid of logarithms :

	A. Total.	B. After absorption of CO <sub>2</sub> .	C. Nitrogen.
Volume of gas ..	3.4670 c.c.	0.2398 c.c.	0.2398 c.c.
Temperature ..	20.6° C.	20.6° C.	20.6° C.
Height of mercury in measuring-tube ..	300 mm.	560 mm.	560 mm.
Height of mercury in pressure-tube ..	258	551	474
Difference ..	42	9	86
Plus tension of aqueous vapour	18	18	18
	60	27	104
Height of barometer ..	767	767	767
	60	27	104
Therefore tension of dry gas ..	707	740	663
Log. of volume of gas ..	0.53995	1.37984	1.37984
0.0012562			
Log. of $(1 + 0.00367 t) 760$ ..	6.18659	6.18659	6.18659
Tension of dry gas ..	2.84942	2.86923	2.82151
Log. of weight of gas calculated as N.	3.57596	4.43566	4.38794
= 0.003767	0.0002727	0.0002443	
0.000273	Deduct blank	0.00008	
			0.00016 N.
	0.003494		
Multiply by 3 ..	= 0.01048		
Divide by 7 ..	= 0.00149		
Deduct blank ..	= 0.0011		
	0.00138	C.	
As 1 litre was used, this represents Carbon ..	0.138 part per 100,000.		
Nitrogen ..	0.016 "		

Ratio of carbon to nitrogen equals 8·6 : 1.

It is necessary that a blank experiment be carried out, using the requisite quantities of all the materials, each time that a fresh supply is bought.

#### Interpretation of Results of Water Analysis.

**Ratio of Organic Carbon to Nitrogen.**—This ratio seems to indicate the nature of the organic matter present. In cases of purely vegetable organic matter the ratio of carbon to nitrogen is high, the reverse being so in that of sewage and other animal contamination. Peaty waters show an average ratio of about 12 : 1 to 20 : 1; surface waters from fields average 6 : 1; shallow wells about 4 : 1; deep wells rather less; sewage 1 : 3.

**Nitrogen as Ammonia.**—The presence of ammonium salts points to recent contamination; their absence may be due to oxidation into nitrates and nitrites. Upland surface waters usually contain about 0·002 part per 100,000; they seldom exceed 0·008. Shallow wells average from 0·1 to 0·15; spring water and deep wells 0·001 to 0·01; sewage from 2 to 10 parts.

A water containing 0·01 part free ammonia per 100,000 should be considered as doubtful.

The amount of albuminoid ammonia in a drinking-water must not exceed 0·008 part per 100,000.

**Oxygen consumed by Organic Matter.**—A very fair conception of the relative purity of a water from organic contamination may be ascertained by estimating the oxygen consumed. Waters contain the following parts per 100,000:

	Great Purity.	Medium Purity.	Doubtfully Pure.	Impure.
Upland surface water ..	Up to 0·1	0·1 to 0·3	0·3 to 0·4	over 0·4
General sources ..	Up to 0·05	0·05 to 0·15	0·15 to 0·2	over 0·2

**Nitrogen as Nitrates and Nitrites.**—These salts are produced by the oxidation of the ammoniacal compounds incident to nitrogenous organic impurities. Where complete oxidation has taken place the nitrogen content exists wholly as nitrates; the presence, therefore, of nitrites shows partial oxidation. The presence of a large amount of nitrates and nitrites shows previous sewage contamination.

Upland surface water .. ..	0·0 to 0·05 part per 100,000.
Surface water from arable soil .. ..	0·25 to 1·0.
Shallow wells .. ..	0·0 to 30·0 (an average of about 5·0).
Deep wells .. ..	0·0 to 5·0 (an average of about 0·5).
Spring water .. ..	Less than deep-well water.

**Chlorine**, unless derived from salt beds or the vicinity of the sea, may always be looked upon as a sure sign of sewage contamination.

Unpolluted waters contain less than 1 part, and no supply should contain more than 2 parts, per 100,000.

Shallow wells may sometimes contain a very large amount.

**Hardness.**—Except for manufacturing and engineering purposes, where deposits, etc., are objected to, hard water does not seem to meet with any objection from a dietetic point of view, only that it is inconvenient from a soap-destroying standpoint. The presence of dissolved salts tends to give the water a pleasant flavour. The exact nature of the hardness depends on the nature of the soil through which the water has passed. Water from shallow wells contains hardnesses varying up to 100 parts or more per 100,000. Pit waters, as a rule, are exceptionally hard. There are various methods of softening water. The temporary hardness in water may be softened by Clark's process, which consists in adding 1 ounce of quicklime for every degree of temporary hardness per 100,000 parts to 700 gallons of the water.

Permanent hardness can only be got rid of by the addition of caustic alkalies, which would be impermissible in a domestic supply.

From a study of the various methods and plants in use, I am of opinion that the plant and process known as the "Criton," manufactured by the Pulsometer Engineering Company, Limited, is by far the most economical and efficient. The same company also make filters capable of filtering both at atmospheric and induced pressures. Suspended matter is of least importance, and can always be got rid of by filtration. The presence of a large amount of organic matter is very inadvisable, as it facilitates the growth of micro-organisms.

## CHAPTER XV

### TYPICAL RESULTS OF ANALYSES

#### Bone Meal.

Moisture .. .. .. .. ..	9'46}	
Organic matter .. .. .. .. ..	28'81}	34'20
Phosphoric acid .. .. .. .. ..	26'05	23'69
Tricalcic phosphate .. .. .. .. ..	56'86	51'71
Lime .. .. .. .. ..	34'65	32'50
Alkalies .. .. .. .. ..	5'31	5'61
Sand .. .. .. .. ..	5'19	4'00
Nitrogen .. .. .. .. ..	3'12	3'25
(Equal to ammonia) .. .. .. .. ..	3'78	3'95

#### Bone Ash (Ground).

Moisture .. .. .. .. ..	0'25	0'40
Organic matter .. .. .. .. ..	0'85	0'80
*Phosphoric acid .. .. .. .. ..	35'50	39'75
Lime .. .. .. .. ..	47'09	52'11
Magnesia, alkalies, etc. .. .. .. .. ..	7'80	4'84
Sand .. .. .. .. ..	8'45	2'10
	<hr/> 100'00	<hr/> 100'00
*Equal to tricalcic phosphate .. .. .. .. ..	77'63	86'78

#### Animal Charcoal (Spent Char).

Carbon .. .. .. .. ..	10'51	
Calcium and magnesium phosphates and fluoride .. .. .. .. ..	80'21	
Calcium carbonate .. .. .. .. ..	8'30	
Calcium sulphate .. .. .. .. ..	0'17	
Ferric oxide .. .. .. .. ..	0'12	
Silica .. .. .. .. ..	0'34	
Alkaline salts .. .. .. .. ..	0'35	
	<hr/> 100'00	

#### Coprolites.

Coprolites are found in England in Bedfordshire, Buckinghamshire, Cambridgeshire, Norfolk and Suffolk; in France and in Russia.

					Per Cent. (Ca <sub>3</sub> P <sub>2</sub> O <sub>10</sub> )
Boulogne	..	..	..	..	38-44
Bellegrade	..	..	..	..	52
Ardennes	..	..	..	..	38-44
Russian	..	..	..	..	46
Cambridge	..	..	..	..	54-60
Bedford	..	..	..	..	50
Welsh	..	..	..	..	62
Silurian Shale	..	..	..	..	56

**Guanos.**

	Moisture.	Organic Matter.	Phosphoric Anhydride.	Lime.	Magnesia, etc.	Silica.	Nitrogen.
Meat meal	10·70	82·23	2·94	2·52	0·90	0·70	10·11
Ichaboc	21·53	38·87	8·46	9·38	8·92	12·86	9·89
Peruvian	19·30	12·55	16·04	14·47	5·64	32·00	2·52
Peruvian	12·62	35·08	14·18	11·59	19·04	7·49	8·87
Malden Island	8·31	3·71	32·86	44·73	10·39	0·00	0·14
Malden Island	7·02	4·08	33·97	46·27	8·66	0·00	0·13
Bats	11·34	21·50	2·83	14·47	17·63	32·23	3·40
Fish guano	23·34	53·67	6·51	8·24	6·47	1·77	7·28

	Nitrogen.	Ammonia
Hair waste	..	13·16
Hair waste	..	11·27
Fur waste	..	11·76
Shoddy	..	7·0

**Phosphates (General).**

	Tricalcic Phosphate (Ca <sub>3</sub> P <sub>2</sub> O <sub>10</sub> ) per Cent.
New Cambridge rock	..
Belgian	..
Ground Canadian	..
Algerian	..
Carolina (mineral)	..
Chisholm Island (mineral)	..
Bordeaux (phosphorite)	..
German (Nassau or Lahn) (phosphorite)	..
Alta Vela (phosphorite)	..
Redonda Island (phosphate of alumina)	..
Sombrero (Carnelian) (phosphate of aluminium)	..
Navassa (phosphate of aluminium)	..
St. Martin's (phosphate of aluminium)	..
Petro Keys (phosphate of aluminium)	..
Aruba (phosphate of aluminium)	..
Canadian (phosphate of aluminium)	..
Belgian (phosphate of aluminium)	..
Bedford (phosphate of aluminium)	..
Port Royal (phosphate of aluminium)	..
Spanish (phosphate of aluminium)	..
Estramadura (phosphate of aluminium)	..

**Soot.**

*Moisture and organic matter	..	..	..	..	73·62
Phosphoric acid	..	..	..	..	0·88
Lime	..	..	..	..	2·32
CO <sub>2</sub> , MgO, Fe <sub>2</sub> O <sub>3</sub> , Al <sub>2</sub> O <sub>3</sub> , etc.	..	..	..	..	5·52
Silica	..	..	..	..	17·66
					100·00

*Nitrogen	..	..	..	..	3·76
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**Cotton Cake (Decorticated).**

Moisture	..	..	10·45	5·15	7·55	10·00
Oil	..	..	11·05	15·55	14·13	8·93
*Albuminoids	..	..	38·43	43·25	44·98	53·46
Mucilage, sugar, etc.	..	..	27·77	26·60	23·43	20·83
Fibre	..	..	4·50	3·20	4·46	1·53
†Ash	..	..	7·80	6·25	5·45	5·25
			100·00	100·00	100·00	100·00
*Nitrogen	..	..	6·10	6·92	7·09	8·47
†Sand	..	..	0·20	0·25	—	0·10

**Cakes (Various).**

		Oil.	Nitrogen.	Fibre.
Cotton cake	..	4·30	3·45	21·0
Decorticated cotton cake	..	16·33	6·65	3·5
Linseed cake	..	18·00	3·65	7·0
Rape cake	..	10·00	—	—
Saccharin cake	..	15·00	3·0	4·0

**Dissolved Bones.**

Moisture	..	..	..	..	..	8·10
'Organic matter	..	..	..	..	..	36·22
Monobasic phosphate	..	..	..	..	..	13·68
(= Tricalcic phosphate rendered soluble)	..	..	..	..	..	21·42
Insoluble phosphates	..	..	..	..	..	10·48
Sulphate of lime, alkalies, etc.	..	..	..	..	..	26·02
Sand	..	..	..	..	..	4·50
						100·00
Nitrogen	..	..	..	..	..	1·90
Equal to ammonia	..	..	..	..	..	2·30

**Kainit.**

Potassium sulphate	..	..	..	..	..	25·51
Calcium sulphate	..	..	..	..	..	1·26
Magnesium sulphate	..	..	..	..	..	18·07
Magnesium chloride	..	..	..	..	..	14·29
Sodium chloride	..	..	..	..	..	25·36
Ferric oxide and alumina	..	..	..	..	..	0·23
Silica	..	..	..	..	..	0·20

## Maize-Meal Cake.

Oil ..	..	..	..	..	..	..	14.90
Albuminoids ..	..	..	..	..	..	..	25.57
(Equal to nitrogen) ..	..	..	..	..	..	..	4.06
Ash ..	..	..	..	..	..	..	5.00

## Corn Manure.

Monobasic phosphate ..	..	..	..	..	..	..	11.95
=Tricalcic phosphate rendered soluble ..	..	..	..	..	..	..	18.71
Insoluble phosphate ..	..	..	..	..	..	..	5.16
Sulphate of lime, alkalies, etc. ..	..	..	..	..	..	..	34.04
Potash ( $K_2O$ ) ..	..	..	..	..	..	..	0.21
Nitrogen ..	..	..	..	..	..	..	3.17

## Linseed Meal.

Moisture ..	..	..	..	..	8.60	10.35
Oil ..	..	..	..	..	32.60	13.03
Albuminoids ..	..	..	..	..	21.10	28.91
Mucilage, sugar, etc. ..	..	..	..	..	26.58	30.23
Woody fibre ..	..	..	..	..	5.17	6.33
Mineral matter ..	..	..	..	..	5.95	5.15
					100.00	100.00
*Nitrogen ..	..	..	..	..	3.36	4.59
†Silica ..	..	..	..	..	1.75	0.25

## Nitrate of Soda.

NaNO <sub>3</sub> ..	..	..	..	..	95.19	97.84
Silica ..	..	..	..	..	0.20	0.12
Moisture ..	..	..	..	..	2.56	1.47
NaCl ..	..	..	..	..	1.60	0.28
Na <sub>2</sub> SO <sub>4</sub> ..	..	..	..	..	0.45	0.29
Refraction ..	..	..	..	..	4.81	2.16

## Muriate of Potash.

Potash ( $K_2O$ ) ..	..	..	..	..	57.42	58.72
=Muriate of potash (KCl) ..	..	..	..	..	90.85	92.93
Moisture ..	..	..	..	..	6.40	4.60

## Bone Manure.

Monobasic phosphate ..	..	..	..	..	..	28.77
Insoluble phosphate ..	..	..	..	..	..	7.16
Nitrogen ..	..	..	..	..	..	0.89

## Sewage Manure.

Phosphoric anhydride ..	..	..	..	..	..	1.58
Lime ..	..	..	..	..	..	8.44
Sulphuric anhydride ..	..	..	..	..	..	4.85
Alumina and ferric oxide ..	..	..	..	..	..	10.94
Magnesia and alkalies ..	..	..	..	..	..	1.13
Nitrogen ..	..	..	..	..	..	1.54

## Turnip Manure.

Soluble phosphate ..	..	..	..	..	..	15.72
Insoluble phosphate ..	..	..	..	..	..	8.77
Nitrogen ..	..	..	..	..	..	1.45

## Fish Manure.

Phosphoric acid ..	..	..	..	..	..	6.88
Lime ..	..	..	..	..	..	12.84
Nitrogen ..	..	..	..	..	..	8.33

## Bean Meal.

Moisture ..	..	..	..	12.75	12.10	7.00	7.20
Oil ..	..	..	..	2.47	1.42	1.66	1.26
*Albuminoids ..	..	..	..	9.73	24.37	26.37	25.51
Carbohydrates ..	..	..	..	56.08	52.35	54.46	51.80
Woody fibre ..	..	..	..	5.50	6.66	6.40	8.33*
†Ash ..	..	..	..	3.47	3.10	4.05	5.90
				100.00	100.00	100.00	100.00
*Containing nitrogen ..	..	..	..	3.16	3.9	4.22	4.05
†Containing sand ..	..	..	..	0.82	0.10	0.85	1.00

## Soils (Various).

Organic matter ..	..	..	..	44.60	38.75	29.64
Sand ..	..	..	..	39.24	35.43	47.56
Lime ..	..	..	..	0.48	0.77	0.48
Iron, alumina, etc.	..	..	..	15.70	22.05	22.32
Nitrogen ..	..	..	..	1.23	1.15	0.94
Potash ..	..	..	..	0.14	0.12	0.05

## Thirds.

Moisture ..	..	..	..	11.80	11.95	12.90
Oil ..	..	..	..	5.67	3.03	3.00
*Albuminoids ..	..	..	..	15.87	15.12	13.18
Mucilage, sugar, etc.	..	..	..	55.31	63.24	63.46
Woody fibre ..	..	..	..	7.00	3.26	3.96
†Ash ..	..	..	..	4.35	2.80	3.50
				100.00	100.00	100.00
*Containing nitrogen ..	..	..	..	2.52	2.48	2.09
†Containing sand ..	..	..	..	0.05	0.15	0.15

## Urine.

Organic matter	..	..	..	..	..	..	77·00*
Nitrogen	..	..	..	..	..	..	8·13
Phosphoric acid	..	..	..	..	..	..	1·23
Lime	..	..	..	..	..	..	1·00

## Superphosphate.

Phosphoric anhydride	..	..	..	..	..	..	12·12
Tricalcic phosphate	..	..	..	..	..	..	26·46
Insoluble phosphates	..	..	..	..	..	..	3·89
Monocalcic phosphate	..	..	..	..	..	..	16·90
Sand	..	..	..	..	..	..	3·99
Organic matter	..	..	..	..	..	..	8·80
Moisture	..	..	..	..	..	..	19·69
Sulphate of lime and alkalies	..	..	..	..	..	..	46·73

## Milk.

AVERAGE COMPOSITION FROM 200,000 ANALYSES (VIETH AND RICHMOND).

Water	..	..	..	..	..	..	87·10
Fat	..	..	..	..	..	..	3·90
Milk-sugar	..	..	..	..	..	..	4·75
Casein	..	..	..	..	..	..	3·00
Albumin	..	..	..	..	..	..	0·40
Ash	..	..	..	..	..	..	0·75

## Cheese (Pearmain and Moor).

Sample.	Water.	Ash.	Fat.	Peichert, C.C. 10 <sup>-3</sup>	Nitrogen.	Casem.
Cheddar	..	33·0	4·3	29·5	4·31	27·4
American	..	29·8	3·7	33·9	4·76	30·3
Gorgonzola	..	40·3	5·3	26·1	4·36	27·7
Dutch	..	41·8	6·3	10·6	27·0	5·11
Gruyère	..	28·2	4·7	28·6	30·0	4·93
Stilton	..	19·4	2·6	42·2	29·0	4·73
Cheshire	..	37·8	4·2	31·3	31·6	4·03
Gloucester	..	33·1	5·0	23·5	31·4	4·99
Camembert	..	47·9	4·7	41·9	31·0	3·43
Parmesan	..	32·5	6·2	17·1	28·0	6·86
Roquefort	..	29·6	6·7	30·3	36·8	4·45
Double cream	..	57·6	3·4	39·3	31·2	3·14
Bondon	..	39·5	0·7	24·4	29·4	1·48
Cream (York)	..	63·1	1·4	6·5	29·0	2·76
						17·9

## Condensed Milk (Pearmain and Moor).

Brand.	Total Solids.	Fat.	Milk- Sugar.	Proteids.	Cane- Sugar.	Remarks.
First Swiss ..	36·7	10·5	14·2	9·7	—	
Ideal ..	38·0	12·4	16·0	8·3	—	Unsweetened
Viking ..	34·2	10·0	13·3	9·0	—	
Anglo-Swiss ..	74·4	10·8	16·0	8·8	37·1	
Fourpenny ..	76·5	10·4	13·0	9·8	41·3	Sweetened
Mother ..	72·0	8·8	13·7	7·3	40·5	whole-milk
Milkmaid ..	76·3	11·0	14·6	9·7	38·7	
Nestlé's ..	77·2	13·7	15·0	9·7	37·2	
Calf ..	58·0	1·0	16·0	7·5	31·9	
Cup ..	56·9	1·0	15·4	8·5	30·4	
Goat ..	71·0	1·2	12·0	9·9	45·9	
Handy ..	75·5	0·3	17·0	12·3	44·3	Sweetened
Lancer ..	67·6	0·3	16·6	12·3	35·8	skim-milk
Minstrel ..	75·3	0·2	15·4	9·7	48·3	

## Kephir.

	König.	Hamerstein.
Fat .. ..	1·44	3·00
Casein .. ..	2·83	2·90
Lactalbumin .. ..	0·36	0·19
Hemi-albumose .. ..	0·26	
Peptone .. ..	0·04	0·07
Sugar .. ..	2·41	2·68
Lactic acid .. ..	1·02	0·73
Ash .. ..	0·68	0·71
Alcohol .. ..	0·75	0·72
Water .. ..	90·21	88·91
	100·00	100·00

## CHAPTER XVI

### TABLES OF FACTORS, ETC.

#### International Atomic Weights.

	O=16.	H=1.		O=16.	H=1.
Aluminium, Al ..	27·1	26·9	Molybdenum, Mo ..	96·0	95·3
Antimony, Sb ..	120·2	119·3	Neodymium, Ne ..	143·6	142·5
Argon, A ..	39·9	39·6	Neon ..	20·0	19·9
Arsenic, As ..	75·0	74·4	Nickel, Ni ..	58·7	58·3
Barium, Ba ..	137·4	136·4	Nitrogen, N ..	14·04	13·93
Bismuth, Bi ..	208·5	206·9	Osmium, Os ..	191·0	189·6
Boron, B ..	11·0	10·9	Oxygen, O ..	16·0	15·88
Bromine, Br ..	79·96	79·36	Palladium, Pd ..	106·5	105·7
Cadmium, Cd ..	112·4	111·6	Phosphorus, P ..	31·0*	30·7*
Cesium, Cs ..	133·0	132·0	Platinum, Pt ..	194·8	193·3
Calcium, Ca ..	40·1	39·8	Potassium, K ..	39·15	38·86
Carbon, C ..	12·00	11·91	Praseodymium, Pr ..	140·5	139·4
Cerium, Ce ..	140·0	139·0	Radium, Ra ..	225·0	223·3
Chlorine, Cl ..	35·45	35·18	Rhodium, Rh ..	103·0	102·2
Chromium, Cr ..	52·1	51·7	Rubidium, Rb ..	85·4	84·8
Cobalt, Co ..	59·0	58·56	Ruthenium, Ru ..	101·7	100·9
Columbium (Nio- bium), Cb ..	94·0	93·3	Samarium, Sm ..	150·0	148·9
Copper, Cu ..	63·6	63·1	Scandium, Sc ..	44·1	43·8
Erbium, E ..	166·0	164·8	Selenium, Se ..	79·2	78·6
Fluorine, F ..	19·0	18·9	Silicon, Si ..	28·4	28·2
Gadolinium, Gd ..	156·0	155·0	Silver, Ag ..	107·93	107·12
Gallium, Ga ..	70·0	69·5	Sodium, Na ..	23·05	22·88
Germanium, Ge ..	72·5	71·9	Strontium, Sr ..	87·6	86·94
Glucinum (Beryl- lium), Gl ..	9·1	9·03	Sulphur, S ..	32·06	31·83
Gold, Au ..	197·2	195·7	Tantalum, Ta ..	183·0	181·6
Helium, He ..	4·0	4·0	Tellurium, Te ..	127·6	126·6
Hydrogen, H ..	1·008	1·000	Terbium, Tb ..	160·0	158·8
Indium, In ..	114·0	113·1	Thallium, Tl ..	204·1	202·6
Iodine, I ..	126·85	125·90	Thorium, Th ..	232·5	230·8
Iridium, Ir ..	193·0	191·5	Thulium, Tm ..	171·0	169·7
Iron, Fe ..	55·9	55·5	Tin, Sn ..	119·0	118·1
Krypton, Kr ..	81·8	81·2	Titanium, Ti ..	48·1	47·7
Lanthanum, La ..	138·9	137·9	Tungsten, W ..	184·0	182·6
Lead, Pb ..	206·9	205·35	Uranium, U ..	238·5	236·7
Lithium, L ..	7·03	6·98	Vanadium, V ..	51·2	50·8
Magnesium, Mg ..	24·36	24·18	Xenon, X* ..	128·0	127·0
Manganese, Mn ..	55·0	54·6	Ytterbium, Yb ..	173·0	171·7
Mercury, Hg ..	200·0	198·5	Yttrium, Yt ..	89·0	88·3
			Zinc, Zn ..	65·4	64·9
			Zirconium, Zr ..	90·6	89·9

Table of Factors.

Required.	Weighed as	Factor.	Logarithm.
Ag	AgCl	0·7527	1·8766273
AgCl	Ag	1·3285	0·1233727
AgBr	Ag	0·5745	1·7592616
AgI	Ag	0·4597	1·6624864
Al	Al <sub>2</sub> O <sub>3</sub>	0·5302	1·7244559
Al	Al <sub>2</sub> P <sub>2</sub> O <sub>8</sub>	0·2218	1·3459667
Al <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> P <sub>2</sub> O <sub>8</sub>	0·4183	1·6215108
As	As <sub>2</sub> S <sub>3</sub>	0·6006	1·7850359
As	Mg <sub>2</sub> As <sub>2</sub> O <sub>7</sub>	0·4840	1·6848231
As <sub>2</sub> O <sub>3</sub>	As <sub>2</sub> S <sub>3</sub>	0·8044	1·9054868
Ba	BaSO <sub>4</sub>	0·5881	1·7694390
BaO	BaSO <sub>4</sub>	0·6567	1·8173625
BaCO <sub>3</sub>	BaSO <sub>4</sub>	0·8454	1·9270469
Bi	Bi <sub>2</sub> O <sub>3</sub>	0·8968	1·9526876
Br	AgBr	1·2350	0·3710621
Ca	CaS	0·7742	1·8906569
Ca	CaO	0·7143	1·8539030
Ca	CaCO <sub>3</sub>	0·4001	1·0021253
CaO*	CaCO <sub>3</sub>	0·5600	1·7482223
C	CO <sub>2</sub>	0·2727	1·4357286
CO <sub>2</sub>	CO <sub>2</sub>	1·3637	0·1346986
CO <sub>2</sub>	CaCO <sub>3</sub>	0·4400	1·6434092
Cl	AgCl	0·2473	1·3932080
CN	AgCN	0·19437	1·28863
Cr <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	0·6882	1·8377090
Cu	CuO	0·7984	1·9022113
Cu	Cu <sub>2</sub> S	0·7980	1·9020468
Cu	Cu <sub>2</sub> O	0·8879	1·9483591
Cu	Cu <sub>2</sub> (CNS) <sub>2</sub>	0·5216	1·7173578
F	CaF <sub>2</sub>	0·4877	1·6881904
Fe	Fe <sub>2</sub> O <sub>3</sub>	0·7002	1·8451913
H	H <sub>2</sub> O	0·11136	1·0467237
HCl	AgCl	0·2543	1·4053162
Hg	HgS	0·8640	1·9365195
H <sub>2</sub> SO <sub>4</sub>	BaSO <sub>4</sub>	0·4205	1·6237806
I	AgI	0·5403	1·7326252
K	K <sub>2</sub> SO <sub>4</sub>	0·4490	1·6522402
K	K <sub>2</sub> PtCl <sub>6</sub>	0·1611	1·2071564
K <sub>2</sub> O	K <sub>2</sub> SO <sub>4</sub>	0·5408	1·7330130
K <sub>2</sub> O	K <sub>2</sub> PtCl <sub>6</sub>	0·1940	1·2879292
K <sub>2</sub> O	KCl	0·6319	1·8006513
KCl	K <sub>2</sub> PtCl <sub>6</sub>	0·3071	1·4872779
Mg	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	0·2166	1·3355931
MgO	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	0·3606	1·5570074
Na	Na <sub>2</sub> SO <sub>4</sub>	0·3243	1·5109565
Na	NaCl	0·3940	1·5954809
Na <sub>2</sub> O	Na <sub>2</sub> SO <sub>4</sub>	0·4368	1·6403344
Na <sub>2</sub> O	NaCl	0·5307	1·7248588

Table of Factors—*continued.*

Required.	Weighed as	Factor.	Logarithm.
N	(NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub>	0·0633	2·8015156
NH <sub>3</sub>	(NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub>	0·0769	2·8857818
NH <sub>4</sub>	(NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub>	0·0814	2·9105912
Pb	PbSO <sub>4</sub>	0·6829	1·8343805
P	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	0·2794	1·4461828
P <sub>2</sub> O <sub>5</sub>	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	0·6394	1·8057830
PO <sub>4</sub>	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	0·8554	1·9321902
Si	SiO <sub>2</sub>	0·4699	1·6720456
Sr	SrSO <sub>4</sub>	0·4767	1·6782784
S	BaSO <sub>4</sub>	0·1375	1·1382314
SO <sub>2</sub>	BaSO <sub>4</sub>	0·2747	1·4388538
SO <sub>3</sub>	BaSO <sub>4</sub>	0·3433	1·5356822
SO <sub>4</sub>	BaSO <sub>4</sub>	0·4119	1·6148091

## Thermometric Scales.

There are two scales most common in use—viz., the Centigrade and Fahrenheit.

$$F^{\circ} = \frac{9}{5} C^{\circ} + 32.$$

$$C^{\circ} = \frac{5}{9} (F^{\circ} - 32).$$

## Relation of Degrees Twadell to Specific Gravity.

$$\text{Degrees Twadell} = \frac{\text{specific gravity} - 1,000}{5}.$$

Specific gravity = (degrees Twadell × 5) + 1,000.  
(Specific gravity of water being equal to 1,000.)

## Conversion of Weights and Measures.

Cubic feet	×	6·2355	= gallons.
Cubic inches	×	0·003607	= gallons.
Ounces	×	28·349	= grammes.
Grains	×	0·0648	= grammes.
Grammes	×	15·432	= grains.
Pints	×	567·936	= cubic centimetres.
Gallons	×	4·548	= litres.
Litres	×	0·22	= gallons.
Centimetres	×	0·3937	= inches.
Inches	×	2·54	= centimetres.
Grains per gallon ÷	0·7		= parts per 100,000.
Parts per 100,000 ×	0·7		= grains per gallon.

One c.c. hydrogen at  $0^{\circ}$  C. and 760 millimetres weighs 0.0000896 grammes.

One c.c. nitrogen at  $0^{\circ}$  C. and 760 millimetres weighs 0.001258 grammes.

One gramme hydrogen at  $0^{\circ}$  C. and 760 millimetres measures 11.6 litres.

Specific gravity of hydrogen, air as unit, = 0.0693.

Specific gravity of air, hydrogen as unit, = 14.43

One c.c. mercury at  $0^{\circ}$  C. weighs 13.596 grammes.

Coefficient of expansion of gases =  $\frac{1}{273} = 0.003665$ .

### Hardness Table (Clark).

PARTS CALCIUM CARBONATE PER 100,000.

C.c. Soap Solution used.	Parts CaCO <sub>3</sub>	C.c. Soap Solution used.	Parts CaCO <sub>3</sub>	C.c. Soap Solution used.	Parts CaCO <sub>3</sub>	C.c. Soap Solution used.	Parts CaCO <sub>3</sub>
0.7	0.00	3.3	3.64	6.0	7.43	8.7	11.35
0.8	0.10	3.4	3.77	6.1	7.57	8.8	11.50
0.9	0.32	3.5	3.90	6.2	7.71	8.9	11.65
1.0	0.48	3.6	4.03	6.3	7.86		
1.1	0.63	3.8	4.29	6.5	8.14	9.1	11.95
1.2	0.79	3.9	4.43	6.6	8.29	9.2	12.11
1.3	0.95			6.7	8.43	9.3	12.26
1.4	1.11	4.0	4.57	6.8	8.57	9.4	12.41
1.5	1.27	4.1	4.71	6.9	8.71	9.5	12.56
1.6	1.43	4.2	4.86			9.6	12.71
1.7	1.50	4.3	5.00	7.0	8.86	9.7	12.86
1.8	1.66	4.4	5.14	7.1	9.00	9.8	13.01
1.9	1.82	4.5	5.29	7.2	9.14	9.9	13.16
		4.6	5.43	7.3	9.29		
2.0	1.95	4.7	5.57	7.4	9.43	10.0	13.31
2.1	2.08	4.8	5.71	7.5	9.57	10.1	13.46
2.2	2.21	4.9	5.86	7.6	9.71	10.2	13.61
2.3	2.34			7.7	9.86	10.3	13.76
2.4	2.47	5.0	6.00	7.8	10.00	10.4	13.91
2.5	2.60	5.1	6.14	7.9	10.15	10.5	14.06
2.6	2.73	5.2	6.29			10.6	14.21
2.7	2.86	5.3	6.43	8.0	10.30	10.7	14.37
2.8	2.99	5.4	6.57	8.1	10.45	10.8	14.52
2.9	3.12	5.5	6.71	8.2	10.60	10.9	14.68
		5.6	6.86	8.3	10.75		
3.0	3.25	5.7	7.00	8.4	10.90	11.0	14.84
3.1	3.38	5.8	7.14	8.5	11.05	11.1	15.00
3.2	3.51	5.9	7.29	8.6	11.20	11.2	15.16

## Hardness Table—continued.

C.c. Soap Solution used.	Parts CaCO <sub>3</sub> .						
11·3	15·32	12·5	17·22	13·7	19·13	14·9	21·03
11·4	15·48	12·6	17·38	13·8	19·29	15·0	21·19
11·5	15·63	12·7	17·54	13·9	19·44	15·1	21·35
11·6	15·79	12·8	17·70	14·0	19·60	15·2	21·51
11·7	15·95	12·9	17·86	14·1	19·76	15·3	21·68
11·8	16·11			14·3	19·92	15·4	21·85
11·9	16·27			14·4	20·08	15·5	22·02
12·0	16·43			14·5	20·24	15·6	22·18
12·1	16·59	13·3	18·49	14·6	20·40	15·7	22·35
12·2	16·75	13·4	18·65	14·7	20·56	15·8	22·52
12·3	16·90	13·5	18·81	14·8	20·71	15·9	22·69
12·4	17·06	13·6	18·97		20·87	16·0	22·86

## Table for Kjeldahl Process (1 Gramme of Substance being used).

One c.c.  $\frac{N}{5}$  acid = 0·0028 grammes N. = 0·0034 grammes NH<sub>3</sub>.

Number of C.c. $\frac{N}{5}$ Acid used.	Per Cent. N.	Per Cent. NH <sub>3</sub> .	Number of C.c. $\frac{N}{5}$ Acid used.	Per Cent. N.	Per Cent. NH <sub>3</sub> .	Number of C.c. $\frac{N}{5}$ Acid used.	Per C.c. N.	Per Cent. NH <sub>3</sub> .
1	0·28	0·34	21	5·88	7·14	41	11·48	13·94
2	0·56	0·68	22	6·16	7·48	42	11·76	14·28
3	0·84	1·02	23	6·44	7·82	43	12·04	14·62
4	1·12	1·36	24	6·72	8·16	44	12·32	14·96
5	1·40	1·70	25	7·00	8·50	45	12·60	15·30
6	1·68	2·04	26	7·28	8·84	46	12·88	15·64
7	1·96	2·38	27	7·56	9·18	47	13·16	15·98
8	2·24	2·72	28	7·84	9·52	48	13·44	16·32
9	2·52	3·06	29	8·12	9·86	49	13·72	16·66
10	2·80	3·40	30	8·40	10·20	50	14·00	17·00
11	3·08	3·74	31	8·68	10·54	51	14·28	17·34
12	3·36	4·08	32	8·96	10·88	52	14·56	17·68
13	3·64	4·42	33	9·24	11·22	53	14·84	18·02
14	3·92	4·76	34	9·52	11·56	54	15·12	18·36
15	4·20	5·10	35	9·80	11·90	55	15·40	18·70
16	4·48	5·44	36	10·08	12·24	56	15·68	19·04
17	4·76	5·78	37	10·36	12·58	57	15·96	19·38
18	5·04	6·12	38	10·64	12·92	58	16·24	19·72
19	5·32	6·46	39	10·92	13·26	59	16·52	20·06
20	5·60	6·80	40	11·20	13·60	60	16·80	20·40

Table for Kjeldahl Process—continued.

C.c. $\frac{N}{5}$ acid used	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9
Per cent. N ...	0·03	0·06	0·08	0·11	0·14	0·17	0·20	0·22	0·25
Per cent. NH <sub>3</sub>	0·03	0·07	0·10	0·14	0·17	0·20	0·24	0·27	0·31

$$\text{Log. } 0\cdot0028 = 3\cdot44716.$$

$$\text{Log. } 0\cdot0034 = 3\cdot53148.$$

Table for converting Nitrogen into Ammonia and Albuminoids.

N.	NH <sub>3</sub> .	Albu-minoiids (N × 6 <sup>25</sup> )	N.	NH <sub>3</sub> .	Albu-minoiids (N × 6 <sup>25</sup> )	N.	NH <sub>3</sub> .	Albu-minoiids (N × 6 <sup>25</sup> )
0·01	0·01	0·06	2·0	2·43	12·50	4·8	5·83	30·00
0·02	0·02	0·13	2·1	2·55	13·13	4·9	5·95	30·63
0·03	0·03	0·19	2·2	2·67	13·75	5·0	6·07	31·25
0·04	0·04	0·25	2·3	2·79	14·38	5·1	6·19	31·88
0·05	0·05	0·31	2·4	2·91	15·00	5·2	6·31	32·50
0·06	0·06	0·38	2·5	3·04	15·63	5·3	6·43	33·13
0·07	0·07	0·44	2·6	3·16	16·25	5·4	6·56	33·75
0·08	0·08	0·50	2·7	3·28	16·88	5·5	6·68	34·38
0·09	0·09	0·56	2·8	3·40	17·50	5·6	6·80	35·00
			2·9	3·52	18·13	5·7	6·92	35·63
0·1	0·12	0·63				5·8	7·04	36·25
0·2	0·24	1·25	3·0	3·64	18·75	5·9	7·16	36·88
0·3	0·36	1·88	3·1	3·76	19·38			
0·4	0·49	2·50	3·2	3·88	20·00	6·0	7·28	37·50
0·5	0·61	3·13	3·3	4·01	20·63	6·1	7·41	38·13
0·6	0·73	3·75	3·4	4·13	21·25	6·2	7·53	38·75
0·7	0·85	4·38	3·5	4·25	21·88	6·3	7·65	39·38
0·8	0·97	5·00	3·6	4·37	22·50	6·4	7·77	40·00
0·9	1·09	5·63	3·7	4·49	23·13	6·5	7·89	40·63
			3·8	4·61	23·75	6·6	8·01	41·25
1·0	1·21	6·25	3·9	4·73	24·38	6·7	8·13	41·88
1·1	1·34	6·88				6·8	8·26	42·50
1·2	1·46	7·50	4·0	4·86	25·00	6·9	8·38	43·13
1·3	1·58	8·13	4·1	4·98	25·63			
1·4	1·70	8·75	4·2	5·10	26·25	7·0	8·50	43·75
1·5	1·82	9·38	4·3	5·22	26·88	7·1	8·62	44·38
1·6	1·94	10·00	4·4	5·34	27·50	7·2	8·74	45·00
1·7	2·06	10·63	4·5	5·46	28·13	7·3	8·86	45·63
1·8	2·19	11·25	4·6	5·58	28·75	7·4	8·98	46·25
1·9	2·31	11·88	4·7	5·71	29·38	7·5	9·11	46·88

Table for converting Nitrogen into Ammonia and  
Albuminoids—continued.

N.	NH <sub>2</sub> .	Albu- minoids (N x 6 <sup>25</sup> )	N.	NH <sub>2</sub> .	Albu- minoids (N x 6 <sup>25</sup> )	N.	NH <sub>2</sub> .	Albu- minoids (N x 6 <sup>25</sup> )
7·6	9·23	47·50	10·4	12·63	65·00	13·2	16·02	82·50
7·7	9·35	48·75	10·5	12·75	65·63	13·3	16·15	83·13
7·8	9·47	48·13	10·6	12·87	66·25	13·4	16·27	83·75
7·9	9·59	49·38	10·7	12·99	66·88	13·5	16·39	84·38
8·0	9·71	50·00	10·9	13·23	67·50	13·6	16·51	85·00
8·1	9·83	50·63				13·7	16·63	85·63
8·2	9·95	51·25	11·0	13·35	68·75	13·8	16·75	86·25
8·3	10·08	51·88	11·1	13·48	69·38	13·9	16·87	86·88
8·4	10·20	52·50	11·2	13·60	70·00	14·0	17·00	87·50
8·5	10·32	53·13	11·3	13·72	70·63	14·1	17·12	88·13
8·6	10·44	53·75	11·4	13·84	71·25	14·2	17·24	88·75
8·7	10·56	54·38	11·5	13·96	71·88	14·3	17·36	89·38
8·8	10·68	55·00	11·6	14·08	72·50	14·4	17·48	90·00
8·9	10·80	55·63	11·7	14·20	73·13	14·5	17·60	90·63
			11·8	14·33	73·75	14·6	17·72	91·25
9·0	10·93	56·25	11·9	14·45	74·38	14·7	17·85	91·88
9·1	11·05	56·88				14·8	17·97	92·50
9·2	11·17	57·50	12·0	14·57	75·00	14·9	18·09	93·13
9·3	11·29	58·13	12·1	14·69	75·63			
9·4	11·41	58·75	12·2	14·81	76·25	15·0	18·21	93·75
9·5	11·53	59·38	12·3	14·93	76·88	15·1	18·33	94·38
9·6	11·65	60·00	12·4	15·05	77·50	15·2	18·45	95·00
9·7	11·78	60·63	12·5	15·18	78·13	15·3	18·57	95·63
9·8	11·90	61·25	12·6	15·30	78·75	15·4	18·70	96·25
9·9	12·02	61·88	12·7	15·42	79·38	15·5	18·82	96·88
			12·8	15·54	80·00	15·6	18·94	97·50
10·0	12·14	62·50	12·9	15·66	80·63	15·7	19·06	98·13
10·1	12·26	63·13				15·8	19·18	98·75
10·2	12·38	63·75	13·0	15·78	81·25	15·9	19·31	99·38
10·3	12·50	64·38	13·1	15·90	81·88	16·0	19·43	100·00

## Soluble or Volatile Acids in Butter Fat.

EXPRESSED AS BUTYRIC ACID, C<sub>4</sub>H<sub>8</sub>O<sub>2</sub>.

C.c. N <sub>10</sub> Alkali.	Per Cent. Soluble Acids.						
1·0	0·18	3·5	0·62	6·0	1·06	8·5	1·50
1·5	0·26	4·0	0·70	6·5	1·14	9·0	1·58
2·0	0·35	4·5	0·79	7·0	1·23	9·5	1·67
2·5	0·44	5·0	0·88	7·5	1·32	10·0	1·76
3·0	0·53	5·5	0·97	8·0	1·41	10·5	1·85

## Soluble or Volatile Acids in Butter Fat—continued.

C.c. N <sub>16</sub> Alkali.	Per Cent. Soluble Acids.						
11°0	1°94	17°0	2°99	23°0	4°05	29°0	5°10
11°5	2°02	17°5	3°08	23°5	4°14	29°5	5°19
12°0	2°11	18°0	3°17	24°0	4°22	30°0	5°28
12°5	2°20	18°5	3°26	24°5	4°31	30°5	5°37
13°0	2°29	19°0	3°34	26°0	4°40	31°0	5°46
13°5	2°38	19°5	3°43	25°5	4°49	31°5	5°54
14°0	2°46	20°0	3°52	26°0	4°58	32°0	5°63
14°5	2°55	20°5	3°61	26°5	4°66	32°5	5°72
15°0	2°64	21°0	3°70	27°0	4°75	33°0	5°81
15°5	2°73	21°5	3°78	27°5	4°84	33°5	5°90
16°0	2°82	22°0	3°87	28°0	4°93	34°0	5°98
16°5	2°90	22°5	3°96	28°5	5°02	34°5	6°07
<hr/>							
C.c. N <sub>10</sub> alkali %.	..	..	0°1	0°2	0°3	0°4	..
Per cent. soluble acids	..	0°02	0°04	0°05	0°07	..	..

## Butter Analysis.

C.c. N <sub>2</sub> Acid used (1 C.c. = 0.008 Gramme KOH).	Solubility KOH required for 1,000 Grammes of Fat (+0°1 C.c. = +0°6).	Saponification Equivalent.	Per Cent. Margarine (+0°1 C.c. = +1°8).
34°9	195°4	286°5	100°0
35°0	196°0	285°7	98°3
35°2	197°1	284°1	94°8
35°4	198°2	282°5	91°3
35°6	199°4	280°9	87°5
35°8	200°5	279°3	84°0
36°0	201°6	277°8	80°5
36°2	202°7	276°2	77°0
36°4	203°8	274°7	73°5
36°6	205°0	273°2	69°7
36°8	206°1	271°7	66°3
37°0	207°2	270°3	62°8
37°2	208°3	268°8	59°3
37°4	209°4	267°4	55°8
37°6	210°6	265°9	52°0
37°8	211°7	264°5	48°5

## Butter Analysis—continued.

C.c. $\frac{1}{2}$ Acid used ( $\times$ C.c. = $\pm 0.028$ Gramme KOH).	Grammes KOH required for 1,000 Grammes of Fat ( $\pm 0.1$ C.c. = $\pm 0.6$ ).	Saponification Equivalent.	Per Cent. Margarine ( $+0.1$ C.c. = $-1.8$ ).
38.0	212.8	263.2	45.0
38.2	213.0	261.8	41.5
38.4	215.0	260.5	38.0
38.6	216.2	259.0	34.2
38.8	217.3	257.7	30.7
39.0	218.4	256.4	27.3
39.2	219.5	255.1	23.8
39.4	220.6	253.9	20.3
39.6	221.8	252.5	16.5
39.8	222.9	251.2	13.0
40.0	224.0	250.0	9.5
40.2	225.1	248.8	6.0
40.4	226.2	247.6	2.5
40.6	227.4	246.8	—
40.8	228.5	245.1	—
41.0	229.6	243.9	—

## Phosphate Table.

Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> .	Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub> .	CaP <sub>2</sub> O <sub>6</sub> .	P <sub>2</sub> O <sub>5</sub> .	P <sub>2</sub> .	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> .	Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub> .	CaP <sub>2</sub> O <sub>6</sub> .	P <sub>2</sub> O <sub>5</sub> .	P <sub>2</sub> .
0.1	0.14	0.09	0.06	0.028	2.0	2.79	1.78	1.28	0.559
0.2	0.28	0.18	0.13	0.056	2.1	2.93	1.87	1.34	0.587
0.3	0.42	0.27	0.19	0.084	2.2	3.07	1.96	1.41	0.614
0.4	0.56	0.36	0.26	0.112	2.3	3.21	2.05	1.47	0.642
0.5	0.70	0.45	0.32	0.140	2.4	3.35	2.14	1.54	0.670
0.6	0.84	0.54	0.38	0.168	2.5	3.49	2.23	1.60	0.698
0.7	0.98	0.62	0.45	0.196	2.6	3.63	2.32	1.66	0.726
0.8	1.12	0.71	0.51	0.223	2.7	3.77	2.41	1.73	0.754
0.9	1.26	0.80	0.58	0.251	2.8	3.91	2.50	1.79	0.782
1.0	1.40	0.89	0.64	0.279	2.9	4.05	2.59	1.86	0.810
1.1	1.54	0.98	0.70	0.307	3.0	4.19	2.68	1.92	0.838
1.2	1.68	1.07	0.77	0.335	3.1	4.33	2.77	1.98	0.866
1.3	1.82	1.16	0.83	0.363	3.2	4.47	2.85	2.05	0.894
1.4	1.96	1.25	0.90	0.391	3.3	4.61	2.94	2.11	0.922
1.5	2.09	1.34	0.96	0.419	3.4	4.75	3.03	2.18	0.950
1.6	2.23	1.43	1.02	0.447	3.5	4.89	3.12	2.24	0.978
1.7	2.37	1.52	1.09	0.475	3.6	5.03	3.21	2.30	1.006
1.8	2.51	1.61	1.15	0.503	3.7	5.17	3.30	2.37	1.033
1.9	2.65	1.70	1.22	0.531	3.8	5.31	3.39	2.43	1.061

## Phosphate Table—continued.

Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub>	Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub>	Ca <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	P <sub>2</sub> O <sub>5</sub>	P <sub>2</sub>	Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub>	Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub>	Ca <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	P <sub>2</sub> O <sub>5</sub>	P <sub>2</sub>
4°0	5·59	3·57	2·56	1·117	9·0	12·57	8·03	5·76	2·514
4°1	5·73	3·66	2·62	1·145	9·1	12·71	8·12	5·82	2·541
4°2	5·87	3·75	2·69	1·173	9·2	12·85	8·21	5·89	2·569
4°3	6·00	3·84	2·75	1·201	9·3	12·99	8·30	5·95	2·597
4°4	6·14	3·93	2·82	1·229	9·4	13·13	8·38	6·01	2·625
4°5	6·28	4·01	2·88	1·257	9·5	13·27	8·47	6·08	2·653
4°6	6·42	4·10	2·94	1·285	9·6	13·41	8·56	6·14	2·681
4°7	6·56	4·19	3·01	1·313	9·7	13·55	8·65	6·21	2·709
4°8	6·70	4·28	3·07	1·341	9·8	13·69	8·74	6·27	2·737
4°9	6·84	4·37	3·14	1·369	9·9	13·83	8·83	6·33	2·765
5°0	6·98	4·46	3·20	1·396	10·0	13·96	8·92	6·40	2·793
5°1	7·12	4·55	3·26	1·424	10·1	14·10	9·01	6·46	2·821
5°2	7·26	4·64	3·33	1·452	10·2	14·24	9·10	6·52	2·849
5°3	7·40	4·73	3·39	1·480	10·3	14·38	9·19	6·59	2·877
5°4	7·54	4·82	3·45	1·508	10·4	14·52	9·28	6·65	2·905
5°5	7·68	4·91	3·52	1·536	10·5	14·66	9·37	6·72	2·932
5°6	7·82	5·00	3·58	1·564	10·6	14·80	9·45	6·78	2·960
5°7	7·96	5·08	3·65	1·592	10·7	14·94	9·54	6·84	2·988
5°8	8·10	5·17	3·71	1·620	10·8	15·08	9·63	6·91	3·016
5°9	8·24	5·26	3·77	1·648	10·9	15·22	9·72	6·97	3·044
6°0	8·38	5·35	3·84	1·676	11·0	15·36	9·81	7·04	3·072
6°1	8·52	5·44	3·90	1·704	11·1	15·50	9·90	7·10	3·100
6°2	8·66	5·53	3·97	1·732	11·2	15·64	9·99	7·16	3·128
6°3	8·80	5·62	4·03	1·760	11·3	15·78	10·08	7·23	3·156
6°4	8·94	5·71	4·09	1·787	11·4	15·92	10·17	7·29	3·184
6°5	9·08	5·80	4·16	1·815	11·5	16·06	10·26	7·36	3·212
6°6	9·22	5·89	4·22	1·843	11·6	16·20	10·35	7·42	3·240
6°7	9·36	5·98	4·29	1·871	11·7	16·34	10·44	7·48	3·268
6°8	9·50	6·07	4·35	1·899	11·8	16·48	10·53	7·55	3·296
6°9	9·64	6·15	4·41	1·927	11·9	16·62	10·61	7·61	3·324
7°0	9·77	6·24	4·48	1·955	12·0	16·76	10·70	7·68	3·351
7°1	9·91	6·33	4·54	1·983	12·1	16·90	10·79	7·74	3·379
7°2	10·05	6·42	4·61	2·011	12·2	17·04	10·88	7·80	3·407
7°3	10·19	6·51	4·67	2·039	12·3	17·18	10·97	7·87	3·435
7°4	10·33	6·60	4·73	2·067	12·4	17·32	11·06	7·93	3·463
7°5	10·47	6·69	4·80	2·095	12·5	17·46	11·15	8·00	3·491
7°6	10·61	6·78	4·86	2·123	12·6	17·60	11·24	8·06	3·519
7°7	10·75	6·87	4·93	2·151	12·7	17·73	11·33	8·12	3·547
7°8	10·89	6·96	4·99	2·178	12·8	17·87	11·42	8·19	3·575
7°9	11·03	7·05	5·05	2·206	12·9	18·01	11·51	8·25	3·603
8°0	11·17	7·14	5·12	2·234	13·0	18·15	11·60	8·32	3·631
8°1	11·31	7·22	5·18	2·262	13·1	18·29	11·68	8·38	3·659
8°2	11·45	7·31	5·25	2·290	13·2	18·43	11·77	8·44	3·687
8°3	11·59	7·40	5·31	2·318	13·3	18·57	11·86	8·51	3·714
8°4	11·73	7·49	5·37	2·346	13·4	18·71	11·95	8·57	3·742
8°5	11·87	7·58	5·44	2·374	13·5	18·85	12·04	8·64	3·770
8°6	12·01	7·67	5·50	2·402	13·6	18·99	12·13	8·70	3·798
8°7	12·15	7·76	5·57	2·430	13·7	19·13	12·22	8·76	3·826
8°8	12·29	7·85	5·63	2·458	13·8	19·27	12·31	8·83	3·854
8°9	12·43	7·94	5·69	2·486	13·9	19·41	12·40	8·89	3·882

## Phosphate Table—continued.

Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	Ca <sub>3</sub> P <sub>2</sub> O <sub>6</sub>	CaP <sub>2</sub> O <sub>6</sub>	P <sub>2</sub> O <sub>5</sub>	P <sub>2</sub>	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	Ca <sub>3</sub> P <sub>2</sub> O <sub>6</sub>	CaP <sub>2</sub> O <sub>6</sub>	P <sub>2</sub> O <sub>5</sub>	P <sub>2</sub>
14°0	19°55	12°49	8°06	3°910	19°0	26°53	16°95	12°15	5°306
14°1	19°69	12°58	9°02	3°938	19°1	26°67	17°04	12°22	5°334
14°2	19°83	12°67	9°08	3°966	19°2	26°81	17°12	12°28	5°362
14°3	19°97	12°76	9°15	3°994	19°3	26°95	17°21	12°35	5°390
14°4	20°11	12°84	9°21	4°022	19°4	27°09	17°30	12°41	5°418
14°5	20°25	12°93	9°28	4°050	19°5	27°23	17°39	12°47	5°446
14°6	20°39	13°02	9°34	4°078	19°6	27°37	17°48	12°54	5°474
14°7	20°53	13°11	9°40	4°105	19°7	27°51	17°57	12°60	5°502
14°8	20°67	13°20	9°47	4°133	19°8	27°65	17°66	12°67	5°530
14°9	20°81	13°29	9°53	4°161	19°9	27°79	17°75	12°73	5°558
15°0	20°95	13°38	9°60	4°189	20°0	27°93	17°84	12°79	5°586
15°1	21°09	13°47	9°66	4°217	20°1	28°07	17°93	12°86	5°614
15°2	21°23	13°56	9°72	4°245	20°2	28°21	18°02	12°92	5°642
15°3	21°37	13°65	9°79	4°273	20°3	28°35	18°11	12°99	5°669
15°4	21°50	13°74	9°85	4°301	20°4	28°49	18°20	13°05	5°697
15°5	21°64	13°83	9°92	4°329	20°5	28°63	18°28	13°11	5°725
15°6	21°78	13°91	9°98	4°357	20°6	28°77	18°37	13°18	5°753
15°7	21°92	14°00	10°04	4°385	20°7	28°91	18°46	13°24	5°781
15°8	22°06	14°09	10°11	4°413	20°8	29°05	18°55	13°31	5°800
15°9	22°20	14°18	10°17	4°441	20°9	29°19	18°64	13°37	5°837
16°0	22°34	14°27	10°23	4°469	21°0	29°32	18°73	13°43	5°865
16°1	22°48	14°36	10°30	4°496	21°1	29°46	18°82	13°50	5°893
16°2	22°62	14°45	10°36	4°524	21°2	29°60	18°91	13°56	5°921
16°3	22°76	14°54	10°43	4°552	21°3	29°74	19°00	13°62	5°949
16°4	22°90	14°63	10°49	4°580	21°4	29°88	19°09	13°66	5°977
16°5	23°04	14°72	10°55	4°608	21°5	30°02	19°18	13°75	6°005
16°6	23°18	14°81	10°62	4°636	21°6	30°16	19°27	13°82	6°033
16°7	23°32	14°89	10°68	4°664	21°7	30°30	19°35	13°88	6°060
16°8	23°46	14°98	10°75	4°692	21°8	30°44	19°44	13°94	6°088
16°9	23°60	15°07	10°81	4°720	21°9	30°58	19°53	14°01	6°116
17°0	23°74	15°16	10°87	4°748	22°0	30°72	19°62	14°07	6°144
17°1	23°88	15°25	10°94	4°776	22°1	30°86	19°71	14°14	6°172
17°2	24°02	15°34	11°00	4°804	22°2	31°00	19°80	14°20	6°200
17°3	24°16	15°43	11°07	4°832	22°3	31°14	19°89	14°26	6°228
17°4	24°30	15°52	11°13	4°860	22°4	31°28	19°98	14°33	6°256
17°5	24°44	15°61	11°19	4°887	22°5	31°42	20°07	14°39	6°284
17°6	24°58	15°70	11°26	4°915	22°6	31°56	20°16	14°46	6°312
17°7	24°72	15°79	11°32	4°943	22°7	31°70	20°25	14°52	6°340
17°8	24°86	15°88	11°39	4°971	22°8	31°84	20°34	14°58	6°368
17°9	25°00	15°97	11°45	4°999	22°9	31°98	20°43	14°65	6°396
18°0	25°14	16°05	11°51	5°027	23°0	32°12	20°51	14°71	6°423
18°1	25°27	16°14	11°58	5°055	23°1	32°26	20°60	14°78	6°451
18°2	25°41	16°23	11°64	5°083	23°2	32°40	20°69	14°84	6°479
18°3	25°55	16°32	11°71	5°111	23°3	32°54	20°78	14°90	6°507
18°4	25°69	16°41	11°77	5°139	23°4	32°68	20°87	14°97	6°535
18°5	25°83	16°50	11°83	5°167	23°5	32°82	20°96	15°03	6°563
18°6	25°97	16°59	11°90	5°195	23°6	32°96	21°05	15°10	6°591
18°7	26°11	16°68	11°96	5°223	23°7	33°09	21°14	15°16	6°619
18°8	26°25	16°77	12°03	5°250	23°8	33°23	21°23	15°22	6°647
18°9	26°39	16°86	12°09	5°278	23°9	33°37	21°32	15°29	6°675

## Phosphate Table—continued.

$Mg_2P_2O_7$	$Ca_3P_2O_8$	$CaP_2O_6$	$P_2O_5$	$P_2$	$Mg_2P_2O_7$	$Ca_3P_2O_8$	$CaP_2O_6$	$P_2O_5$	$P_2$
24°0	33°51	21°41	15°35	6°703	29°0	40°50	25°87	18°55	8°099
24°1	33°65	21°50	15°42	6°731	29°1	40°64	25°95	18°61	8°127
24°2	33°79	21°58	15°48	6°759	29°2	40°78	26°04	18°68	8°155
24°3	33°93	21°67	15°54	6°787	29°3	40°92	26°13	18°74	8°183
24°4	34°07	21°76	15°61	6°814	29°4	41°06	26°22	18°81	8°211
24°5	34°21	21°85	15°67	6°842	29°5	41°19	26°31	18°87	8°239
24°6	34°35	21°94	15°74	6°870	29°6	41°33	26°40	18°93	8°267
24°7	34°49	22°03	15°80	6°898	29°7	41°47	26°49	19°00	8°295
24°8	34°63	22°12	15°86	6°926	29°8	41°61	26°58	19°06	8°323
24°9	34°77	22°21	15°93	6°954	29°9	41°75	26°67	19°13	8°351
25°0	34°91	22°30	15°99	6°982	30°0	41°89	26°76	19°19	8°378
25°1	35°05	22°39	16°06	7°010	30°1	42°03	26°85	19°25	8°406
25°2	35°19	22°48	16°12	7°038	30°2	42°17	26°94	19°32	8°434
25°3	35°33	22°57	16°18	7°066	30°3	42°31	27°03	19°38	8°462
25°4	35°47	22°66	16°25	7°094	30°4	42°45	27°11	19°45	8°490
25°5	35°61	22°74	16°31	7°122	30°5	42°59	27°20	19°51	8°518
25°6	35°75	22°83	16°38	7°150	30°6	42°73	27°29	19°57	8°546
25°7	35°89	22°92	16°44	7°178	30°7	42°87	27°38	19°64	8°574
25°8	36°03	23°01	16°50	7°205	30°8	43°01	27°47	19°70	8°602
25°9	36°17	23°10	16°57	7°233	30°9	43°15	27°56	19°77	8°630
26°0	36°31	23°19	16°63	7°261	31°0	43°29	27°65	19°83	8°658
26°1	36°45	23°28	16°70	7°289	31°1	43°43	27°74	19°89	8°686
26°2	36°59	23°37	16°76	7°317	31°2	43°57	27°83	19°96	8°714
26°3	36°73	23°46	16°82	7°345	31°3	43°71	27°92	20°02	8°742
26°4	36°87	23°55	16°89	7°373	31°4	43°85	28°01	20°09	8°769
26°5	37°00	23°64	16°95	7°401	31°5	43°99	28°10	20°15	8°797
26°6	37°14	23°72	17°02	7°429	31°6	44°13	28°18	21°21	8°825
26°7	37°28	23°81	17°08	7°457	31°7	44°27	28°27	20°28	8°853
26°8	37°42	23°90	17°14	7°485	31°8	44°41	28°36	20°34	8°881
26°9	37°56	23°99	17°21	7°513	31°9	44°55	28°45	20°41	8°909
27°0	37°70	24°08	17°27	7°541	32°0	44°69	28°54	20°47	8°937
27°1	37°84	24°17	17°33	7°569	32°1	44°82	28°63	20°53	8°965
27°2	37°98	24°26	17°40	7°597	32°2	44°96	28°72	20°60	8°993
27°3	38°12	24°35	17°46	7°624	32°3	45°10	28°81	20°69	9°021
27°4	38°26	24°44	17°53	7°652	32°4	45°24	28°90	20°72	9°049
27°5	38°40	24°53	17°59	7°680	32°5	45°38	28°99	20°79	9°077
27°6	38°54	24°62	17°65	7°708	32°6	45°52	29°08	20°85	9°105
27°7	38°68	24°71	17°72	7°736	32°7	45°66	29°17	20°92	9°133
27°8	38°82	24°80	17°78	7°764	32°8	45°80	29°26	20°98	9°160
27°9	38°96	24°88	17°85	7°792	32°9	45°94	29°34	21°04	9°188
28°0	39°10	24°97	17°91	7°820	33°0	46°08	29°43	21°11	9°216
28°1	39°24	25°06	17°97	7°848	33°1	46°22	29°52	21°17	9°244
28°2	39°38	25°15	18°04	7°876	33°2	46°36	29°61	21°24	9°262
28°3	39°52	25°24	18°10	7°904	33°3	46°50	29°70	21°30	9°300
28°4	39°66	25°33	18°17	7°932	33°4	46°64	29°79	21°36	9°328
28°5	39°80	25°42	18°23	7°959	33°5	46°78	29°88	21°43	9°356
28°6	39°94	25°51	18°29	7°987	33°6	46°92	29°97	21°49	9°384
28°7	40°08	25°60	18°36	8°015	33°7	47°06	30°06	21°56	9°412
28°8	40°22	25°69	18°42	8°043	33°8	47°20	30°15	21°62	9°440
28°9	40°36	25°78	18°49	8°071	33°9	47°34	30°24	21°68	9°468

## Phosphate Table—continued.

Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub>	CaP <sub>2</sub> O <sub>8</sub>	P <sub>2</sub> O <sub>5</sub>	P <sub>2</sub>	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub>	CaP <sub>2</sub> O <sub>8</sub>	P <sub>2</sub> O <sub>5</sub>	• P <sub>2</sub>
34°0	47°48	30°33	21°75	9°496	39°0	54°46	34°78	24°95	10°892
34°1	47°62	30°41	21°81	9°523	39°1	54°60	34°87	25°01	10°920
34°2	47°76	30°50	21°88	9°551	39°2	54°74	34°96	25°07	10°948
34°3	47°90	30°59	21°94	9°579	39°3	54°88	35°05	25°14	10°976
34°4	48°04	30°68	22°00	9°607	39°4	55°02	35°14	25°20	11°004
34°5	48°18	30°77	22°07	9°635	39°5	55°16	35°23	25°27	11°032
34°6	48°32	30°86	22°13	9°663	39°6	55°30	35°32	25°33	11°060
34°7	48°46	30°95	22°20	9°691	39°7	55°44	35°41	25°39	11°087
34°8	48°60	31°04	22°26	9°719	39°8	55°58	35°50	25°46	11°115
34°9	48°74	31°13	22°32	9°747	39°9	55°72	35°59	25°52	11°143
35°0	48°87	31°22	22°39	9°775	40°0	55°86	35°68	25°59	11°171
35°1	49°01	31°31	22°45	9°803	40°1	56°00	35°77	25°65	11°199
35°2	49°15	31°40	22°52	9°831	40°2	56°14	35°85	25°71	11°227
35°3	49°29	31°49	22°58	9°859	40°3	56°28	35°94	25°78	11°255
35°4	49°43	31°57	22°64	9°887	40°4	56°42	36°03	25°84	11°283
35°5	49°57	31°66	22°71	9°914	40°5	56°55	36°12	25°91	11°311
35°6	49°71	31°75	22°77	9°942	40°6	56°69	36°21	25°97	11°339
35°7	49°85	31°84	22°84	9°970	40°7	56°83	36°30	26°03	11°367
35°8	49°99	31°93	22°90	9°998	40°8	56°97	36°39	26°10	11°395
35°9	50°13	32°02	22°96	10°026	40°9	57°11	36°48	26°16	11°423
36°0	50°27	32°11	23°03	10°054	41°0	57°25	36°57	26°23	11°451
36°1	50°41	32°20	23°09	10°082	41°1	57°39	36°66	26°29	11°478
36°2	50°55	32°29	23°16	10°110	41°2	57°53	36°75	26°35	11°506
36°3	50°69	32°38	23°22	10°138	41°3	57°67	36°84	26°42	11°534
36°4	50°83	32°47	23°28	10°166	41°4	57°81	36°93	26°48	11°562
36°5	50°97	32°55	23°35	10°194	41°5	57°95	37°01	26°55	11°590
36°6	51°11	32°64	23°41	10°222	41°6	58°09	37°10	26°61	11°618
36°7	51°25	32°73	23°48	10°250	41°7	58°23	37°19	26°67	11°646
36°8	51°39	32°82	23°54	10°278	41°8	58°37	37°28	26°74	11°674
36°9	51°53	32°91	23°60	10°306	41°9	58°51	37°37	26°80	11°702
37°0	51°67	33°00	23°67	10°333	42°0	58°65	37°46	26°87	11°730
37°1	51°81	33°09	23°73	10°361	42°1	58°79	37°55	26°93	11°758
37°2	51°95	33°18	23°80	10°389	42°2	58°93	37°64	26°99	11°786
37°3	52°09	33°27	23°86	10°417	42°3	59°07	37°73	27°06	11°814
37°4	52°23	33°36	23°92	10°445	42°4	59°21	37°82	27°12	11°842
37°5	52°37	33°45	23°99	10°473	42°5	59°35	37°91	27°19	11°869
37°6	52°51	33°54	24°05	10°501	42°6	59°49	38°00	27°25	11°897
37°7	52°64	33°62	24°12	10°529	42°7	59°63	38°08	27°31	11°925
37°8	52°78	33°71	24°18	10°557	42°8	59°77	38°17	27°38	11°953
37°9	52°92	33°80	24°24	10°585	42°9	59°91	38°26	27°44	11°981
38°0	53°06	33°89	24°31	10°613	43°0	60°05	38°35	27°51	12°009
38°1	53°20	33°98	24°37	10°641	43°1	60°18	38°44	27°57	12°037
38°2	53°34	34°07	24°43	10°669	43°2	60°32	38°53	27°63	12°065
38°3	53°48	34°16	24°50	10°696	43°3	60°46	38°62	27°70	12°093
38°4	53°62	34°25	24°56	10°724	43°4	60°60	38°71	27°76	12°121
38°5	53°76	34°34	24°63	10°752	43°5	60°74	38°80	27°83	12°149
38°6	53°90	34°43	24°69	10°780	43°6	60°88	38°89	27°89	12°177
38°7	54°04	34°52	24°75	10°808	43°7	61°02	38°98	27°95	12°205
38°8	54°18	34°61	24°82	10°836	43°8	61°16	39°07	28°02	12°232
38°9	54°32	34°70	24°88	10°864	43°9	61°30	39°16	28°08	12°260

Phosphate Table—*continued*

Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub>	CaP <sub>2</sub> O <sub>6</sub>	P <sub>2</sub> O <sub>5</sub>	P <sub>2</sub>	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub>	CaP <sub>2</sub> O <sub>6</sub>	P <sub>2</sub> O <sub>5</sub>	P <sub>2</sub>
44°0	61°44	39°24	28°14	12°288	49°0	68°42	43°70	31°34	13°685
44°1	61°58	39°33	28°21	12°316	49°1	68°56	43°79	31°41	13°713
44°2	61°72	39°42	28°27	12°344	49°2	68°70	43°88	31°47	13°741
44°3	61°86	39°51	28°34	12°372	49°3	68°84	43°97	31°53	13°769
44°4	62°00	39°60	28°40	12°400	49°4	68°98	44°06	31°60	13°796
44°5	62°14	39°69	28°46	12°428	49°5	69°12	44°15	31°66	13°824
44°6	62°28	39°78	28°53	12°456	49°6	69°26	44°24	31°73	13°852
44°7	62°42	39°87	28°59	12°484	49°7	69°40	44°33	31°79	13°880
44°8	62°56	39°96	28°66	12°512	49°8	69°54	44°22	31°85	13°908
44°9	62°70	40°05	28°72	12°540	49°9	69°68	44°51	31°92	13°936
45°0	62°84	40°14	28°78	12°568	50°0	69°82	44°60	31°98	13°964
45°1	62°98	40°23	28°85	12°596	50°1	69°96	44°68	32°05	13°992
45°2	63°12	40°31	28°91	12°624	50°2	70°10	44°77	32°11	14°020
45°3	63°26	40°40	28°98	12°651	50°3	70°24	44°86	32°17	14°048
45°4	63°40	40°49	29°04	12°679	50°4	70°38	44°95	32°24	14°076
45°5	63°54	40°58	29°10	12°707	50°5	70°52	45°04	32°30	14°104
45°6	63°68	40°67	29°17	12°735	50°6	70°66	45°13	32°37	14°132
45°7	63°82	40°76	29°23	12°763	50°7	70°80	45°22	32°43	14°160
45°8	63°96	40°85	29°30	12°791	50°8	70°94	45°31	32°49	14°187
45°9	64°10	40°94	29°36	12°819	50°9	71°08	45°40	32°56	14°215
46°0	64°23	41°03	29°42	12°847	51°0	71°22	45°49	32°62	14°243
46°1	64°37	41°12	29°49	12°875	51°1	71°36	45°58	32°69	14°271
46°2	64°51	41°21	29°55	12°903	51°2	71°50	45°67	32°75	14°299
46°3	64°65	41°30	29°62	12°931	51°3	71°64	45°76	32°81	14°327
46°4	64°79	41°38	29°68	12°959	51°4	71°78	45°84	32°88	14°355
46°5	64°93	41°47	29°74	12°987	51°5	71°91	45°93	32°94	14°383
46°6	65°07	41°56	29°81	13°015	51°6	72°05	46°02	33°01	14°411
46°7	65°21	41°65	29°87	13°042	51°7	72°19	46°11	33°07	14°439
46°8	65°35	41°74	29°94	13°070	51°8	72°33	46°20	33°13	14°467
46°9	65°49	41°83	30°00	13°098	51°9	72°47	46°29	33°20	14°495
47°0	65°63	41°92	30°06	13°126	52°0	72°61	46°38	33°26	14°523
47°1	65°77	42°01	30°13	13°154	52°1	72°75	46°47	33°33	14°551
47°2	65°91	42°10	30°19	13°182	52°2	72°89	46°56	33°39	14°579
47°3	66°05	42°19	30°26	13°210	52°3	73°03	46°65	33°45	14°606
47°4	66°19	42°28	30°32	13°238	52°4	73°17	46°74	33°52	14°634
47°5	66°33	42°37	30°38	13°266	52°5	73°31	46°83	33°58	14°662
47°6	66°47	42°45	30°45	13°294	52°6	73°45	46°91	33°65	14°690
47°7	66°61	42°54	30°51	13°322	52°7	73°59	47°00	33°71	14°718
47°8	66°75	42°63	30°58	13°350	52°8	73°73	47°09	33°77	14°746
47°9	66°89	42°72	30°64	13°378	52°9	73°87	47°18	33°84	14°774
48°0	67°03	42°81	30°70	13°405	53°0	74°01	47°27	33°90	14°802
48°1	67°17	42°90	30°77	13°433	53°1	74°15	47°36	33°97	14°830
48°2	67°31	42°99	30°83	13°461	53°2	74°29	47°45	34°03	14°868
48°3	67°45	43°08	30°90	13°489	53°3	74°43	47°54	34°09	14°886
48°4	67°59	43°17	30°96	13°517	53°4	74°57	47°63	34°16	14°914
48°5	67°73	43°26	31°02	13°545	53°5	74°71	47°72	34°22	14°941
48°6	67°87	43°35	31°09	13°573	53°6	74°85	47°81	34°29	14°969
48°7	68°00	43°44	31°15	13°601	53°7	74°99	47°90	34°35	14°997
48°8	68°14	43°53	31°22	13°629	53°8	75°13	47°99	34°41	15°025
48°9	68°28	43°61	31°28	13°657	53°9	75°27	48°07	34°48	15°053

## Phosphate Table—continued.

$\bullet M_{K_2}P_2O_7$	$Ca_3P_2O_8$	$Ca_1P_2O_6$	$P_2O_5$	$P_2$	$Mg_2P_2O_7$	$Ca_3P_2O_8$	$Ca_1P_2O_6$	$P_2O_5$	$\bullet P_2$
54°0	75°41	48°16	34°54	15°081	58°0	80°99	51°73	37°10	16°198
54°1	75°55	48°25	34°61	15°109	58°1	81°13	51°82	37°16	16°226
54°2	75°69	48°34	34°67	15°137	58°2	81°27	51°91	37°23	16°254
54°3	75°83	48°43	34°73	15°165	58°3	81°41	52°00	37°29	16°282
54°4	75°97	48°52	34°80	15°193	58°4	81°55	52°09	37°36	16°310
54°5	76°10	48°61	34°86	15°221	58°5	81°69	52°18	37°42	16°338
54°6	76°24	48°70	34°93	15°249	58°6	81°83	52°27	37°48	16°366
54°7	76°38	48°79	34°99	15°277	58°7	81°97	52°35	37°55	16°394
54°8	76°52	48°88	35°05	15°305	58°8	82°11	52°44	37°61	16°422
54°9	76°66	48°97	35°12	15°333	58°9	82°25	52°53	37°68	16°450
55°0	76°80	49°06	35°18	15°360	59°0	82°39	52°62	37°74	16°478
55°1	76°94	49°14	35°24	15°388	59°1	82°53	52°71	37°80	16°505
55°2	77°08	49°23	35°31	15°416	59°2	82°67	52°80	37°87	16°533
55°3	77°22	49°32	35°37	15°444	59°3	82°81	52°89	37°93	16°561
55°4	77°36	49°41	35°44	15°472	59°4	82°95	52°98	38°00	16°589
55°5	77°50	49°50	35°50	15°500	59°5	83°09	53°07	38°06	16°617
55°6	77°64	49°59	35°56	15°528	59°6	83°23	53°16	38°12	16°645
55°7	77°78	49°68	35°63	15°556	59°7	83°37	53°25	38°19	16°673
55°8	77°92	49°77	35°69	15°584	59°8	83°51	53°34	38°25	16°701
55°9	78°06	49°86	35°76	15°612	59°9	83°65	53°43	38°32	16°729
56°0	78°20	49°95	35°82	15°640	60°0	83°78	53°51	38°38	16°757
56°1	78°34	49°103	35°88	15°668	60°1	83°92	53°60	38°44	16°785
56°2	78°48	50°12	35°95	15°696	60°2	84°00	53°69	38°51	16°813
56°3	78°62	50°21	36°01	15°724	60°3	84°20	53°78	38°57	16°841
56°4	78°76	50°30	36°08	15°751	60°4	84°34	53°87	38°63	16°869
56°5	78°90	50°39	36°14	15°779	60°5	84°48	53°96	38°70	16°896
56°6	79°04	50°48	36°20	15°807	60°6	84°62	54°05	38°76	16°924
56°7	79°18	50°57	36°27	15°835	60°7	84°76	54°14	38°83	16°952
56°8	79°32	50°66	36°33	15°863	60°8	84°90	54°23	38°89	16°980
56°9	79°46	50°75	36°40	15°891	60°9	85°04	54°32	38°95	17°008
57°0	79°60	50°84	36°46	15°919	61°0	85°18	54°41	39°02	17°036
57°1	79°74	50°93	36°52	15°947	62°0	86°58	55°30	39°66	17°315
57°2	79°87	51°02	36°59	15°975	63°0	87°97	56°19	40°30	17°595
57°3	80°01	51°11	36°65	16°003	64°0	89°37	57°08	40°94	17°874
57°4	80°15	51°20	36°72	16°031	65°0	90°77	57°97	41°58	18°153
57°5	80°29	51°28	36°78	16°059	66°0	92°16	58°87	42°22	18°433
57°6	80°43	51°37	36°84	16°087	67°0	93°56	59°76	42°86	18°712
57°7	80°57	51°46	36°91	16°115	68°0	94°96	60°65	43°50	18°991
57°8	80°71	51°55	36°97	16°142	69°0	96°35	61°54	44°14	19°270
57°9	80°85	51°64	37°04	16°170	70°0	97°75	62°43	44°78	19°550
					71°0	99°14	63°33	45°41	19°829
						100°00	63°87	45°81	20°000

$Mg_2P_2O_7$	0°010	0°020	0°030	0°040	0°050	0°060	0°070	0°080	0°090
$Ca_3P_2O_8$	0°010	0°030	0°040	0°060	0°070	0°080	0°100	0°110	0°130
$Ca_1P_2O_6$	0°010	0°020	0°030	0°040	0°050	0°050	0°060	0°070	0°080
$P_2O_5$	0°010	0°010	0°020	0°030	0°040	0°040	0°050	0°050	0°060
$P_2$	0°0036	0°006	0°008	0°011	0°017	0°017	0°020	0°022	0°025

## Reduction of G.c. Nitrogen to Grammes.

$$\text{Log}_{\frac{0.0012562}{(1+0.003674)760}} \text{for each } i_v^\circ \text{ C. from } 0^\circ \text{ to } 30^\circ \text{ C.}$$

Temperature C.	$0^\circ$	$0.1^\circ$	$0.2^\circ$	$0.3^\circ$	$0.4^\circ$	$0.5^\circ$	$0.6^\circ$	$0.7^\circ$	$0.8^\circ$	$0.9^\circ$
0°	6.21824	808	793	777	761	*745	729	713	697	681
1°	6.21665	649	633	617	601	586	570	554	538	522
2°	6.21597	491	475	459	443	427	412	396	380	364
3°	6.21349	333	318	302	286	270	255	239	223	208
4°	6.21192	177	161	145	130	114	098	083	067	051
5°	6.21035	020	004	*989	*973	*957	*942	*926	*911	*895
6°	6.20879	864	848	833	817	801	786	770	755	739
7°	6.20723	708	692	676	661	645	629	614	598	583
8°	6.20567	552	536	521	505	490	475	459	443	428
9°	6.20413	397	382	366	351	335	320	304	289	274
10°	6.20259	244	228	213	198	182	167	151	136	121
11°	6.20106	090	075	060	045	029	014	*999	*984	*969
12°	6.19953	938	923	907	892	877	862	846	831	816
13°	6.19880	785	770	755	740	724	709	694	679	664
14°	6.19648	633	618	603	588	573	558	543	528	513
15°	6.19497	482	467	452	437	422	407	392	377	362
16°	6.19346	331	316	301	286	271	256	392	226	211
17°	6.19196	181	166	157	136	121	106	091	076	061
18°	6.19046	031	016	001	*986	*971	*956	*941	*926	*911
19°	6.18897	*882	867	852	837	822	807	792	777	762
20°	6.18748	733	718	703	688	673	659	644	629	614
21°	6.18600	585	570	555	540	526	511	496	481	466
22°	6.18452	437	422	408	393	378	363	349	334	319
23°	6.18305	290	*275	261	246	231	216	202	187	172
24°	6.18158	143	128	114	099	084	070	055	041	026
25°	6.18012	*997	*982	*968	*953	*938	*924	*909	*895	*880
26°	6.17866	851	837	822	808	793	779	764	750	735
27°	6.17721	706	692	677	663	648	634	619	605	590
28°	6.17576	561	547	532	518	503	489	475	460	446
29°	6.17432	417	393	388	374	360	345	331	316	302

## Elasticity of Aqueous Vapour

FOR EACH  $\frac{1}{5}^{\circ}$  C. FROM  $0^{\circ}$  TO  $30^{\circ}$  (REGNAULT).

Temper- ature C.	Tension in Min. Mercury.	Temper- ature C.	Tension in Min. Mercury.	Temper- ature C.	Tension in Min. Mercury.	Temper- ature C.	Tension in Min. Mercury.	Temper- ature C.	Tension in Min. Mercury.
$0^{\circ}$	4·6	$4^{\circ}0^{\circ}$	6·1	$8^{\circ}0^{\circ}$	8·0	$12^{\circ}0^{\circ}$	10·5	$16^{\circ}0^{\circ}$	13·5
$0\cdot1^{\circ}$	4·6	$4^{\circ}1^{\circ}$	6·1	$8^{\circ}1^{\circ}$	8·1	$12^{\circ}1^{\circ}$	10·5	$16^{\circ}1^{\circ}$	13·6
$0\cdot2^{\circ}$	4·7	$4^{\circ}2^{\circ}$	6·2	$8^{\circ}2^{\circ}$	8·1	$12^{\circ}2^{\circ}$	10·6	$16^{\circ}2^{\circ}$	13·7
$0\cdot3^{\circ}$	4·8	$4^{\circ}3^{\circ}$	6·2	$8^{\circ}3^{\circ}$	8·2	$12^{\circ}3^{\circ}$	10·7	$16^{\circ}3^{\circ}$	13·8
$0\cdot4^{\circ}$	4·7	$4^{\circ}4^{\circ}$	6·3	$8^{\circ}4^{\circ}$	8·2	$12^{\circ}4^{\circ}$	10·7	$16^{\circ}4^{\circ}$	13·9
$0\cdot5^{\circ}$	4·8	$4^{\circ}5^{\circ}$	6·3	$8^{\circ}5^{\circ}$	8·3	$12^{\circ}5^{\circ}$	10·8	$16^{\circ}5^{\circ}$	14·0
$0\cdot6^{\circ}$	4·8	$4^{\circ}6^{\circ}$	6·4	$8^{\circ}6^{\circ}$	8·3	$12^{\circ}6^{\circ}$	10·9	$16^{\circ}6^{\circ}$	14·1
$0\cdot7^{\circ}$	4·8	$4^{\circ}7^{\circ}$	6·4	$8^{\circ}7^{\circ}$	8·4	$12^{\circ}7^{\circ}$	10·9	$16^{\circ}7^{\circ}$	14·2
$0\cdot8^{\circ}$	4·9	$4^{\circ}8^{\circ}$	6·4	$8^{\circ}8^{\circ}$	8·5	$12^{\circ}8^{\circ}$	11·0	$16^{\circ}8^{\circ}$	14·2
$0\cdot9^{\circ}$	4·9	$4^{\circ}9^{\circ}$	6·5	$8^{\circ}9^{\circ}$	8·5	$12^{\circ}9^{\circ}$	11·1	$16^{\circ}9^{\circ}$	14·3
$1\cdot0^{\circ}$	4·9	$5^{\circ}0^{\circ}$	6·5	$9^{\circ}0^{\circ}$	8·6	$13^{\circ}0^{\circ}$	11·2	$17^{\circ}0^{\circ}$	14·4
$1\cdot1^{\circ}$	5·0	$5^{\circ}1^{\circ}$	6·6	$9^{\circ}1^{\circ}$	8·6	$13^{\circ}1^{\circ}$	11·2	$17^{\circ}1^{\circ}$	14·5
$1\cdot2^{\circ}$	5·0	$5^{\circ}2^{\circ}$	6·6	$9^{\circ}2^{\circ}$	8·7	$13^{\circ}2^{\circ}$	11·3	$17^{\circ}2^{\circ}$	14·6
$1\cdot3^{\circ}$	5·0	$5^{\circ}3^{\circ}$	6·7	$9^{\circ}3^{\circ}$	8·7	$13^{\circ}3^{\circ}$	11·4	$17^{\circ}3^{\circ}$	14·7
$1\cdot4^{\circ}$	5·1	$5^{\circ}4^{\circ}$	6·7	$9^{\circ}4^{\circ}$	8·8	$13^{\circ}4^{\circ}$	11·5	$17^{\circ}4^{\circ}$	14·8
$1\cdot5^{\circ}$	5·1	$5^{\circ}5^{\circ}$	6·8	$9^{\circ}5^{\circ}$	8·9	$13^{\circ}5^{\circ}$	11·5	$17^{\circ}5^{\circ}$	14·9
$1\cdot6^{\circ}$	5·2	$5^{\circ}6^{\circ}$	6·8	$9^{\circ}6^{\circ}$	8·9	$13^{\circ}6^{\circ}$	11·6	$17^{\circ}6^{\circ}$	15·0
$1\cdot7^{\circ}$	5·2	$5^{\circ}7^{\circ}$	6·9	$9^{\circ}7^{\circ}$	9·0	$13^{\circ}7^{\circ}$	11·7	$17^{\circ}7^{\circ}$	15·1
$1\cdot8^{\circ}$	5·2	$5^{\circ}8^{\circ}$	6·9	$9^{\circ}8^{\circ}$	9·0	$13^{\circ}8^{\circ}$	11·8	$17^{\circ}8^{\circ}$	15·2
$1\cdot9^{\circ}$	5·3	$5^{\circ}9^{\circ}$	7·0	$9^{\circ}9^{\circ}$	9·1	$13^{\circ}9^{\circ}$	11·8	$17^{\circ}9^{\circ}$	15·3
$2\cdot0^{\circ}$	5·3	$6^{\circ}0^{\circ}$	7·0	$10^{\circ}0^{\circ}$	9·2	$14^{\circ}0^{\circ}$	11·9	$18^{\circ}0^{\circ}$	15·4
$2\cdot1^{\circ}$	5·3	$6^{\circ}1^{\circ}$	7·0	$10^{\circ}1^{\circ}$	9·2	$14^{\circ}1^{\circ}$	12·0	$18^{\circ}1^{\circ}$	15·5
$2\cdot2^{\circ}$	5·4	$6^{\circ}2^{\circ}$	7·1	$10^{\circ}2^{\circ}$	9·3	$14^{\circ}2^{\circ}$	12·1	$18^{\circ}2^{\circ}$	15·6
$2\cdot3^{\circ}$	5·4	$6^{\circ}3^{\circ}$	7·1	$10^{\circ}3^{\circ}$	9·3	$14^{\circ}3^{\circ}$	12·1	$18^{\circ}3^{\circ}$	15·7
$2\cdot4^{\circ}$	5·5	$6^{\circ}4^{\circ}$	7·2	$10^{\circ}4^{\circ}$	9·4	$14^{\circ}4^{\circ}$	12·2	$18^{\circ}4^{\circ}$	15·7
$2\cdot5^{\circ}$	5·5	$6^{\circ}5^{\circ}$	7·2	$10^{\circ}5^{\circ}$	9·5	$14^{\circ}5^{\circ}$	12·3	$18^{\circ}5^{\circ}$	15·8
$2\cdot6^{\circ}$	5·5	$6^{\circ}6^{\circ}$	7·3	$10^{\circ}6^{\circ}$	9·5	$14^{\circ}6^{\circ}$	12·4	$18^{\circ}6^{\circ}$	15·9
$2\cdot7^{\circ}$	5·6	$6^{\circ}7^{\circ}$	7·3	$10^{\circ}7^{\circ}$	9·6	$14^{\circ}7^{\circ}$	12·5	$18^{\circ}7^{\circ}$	16·0
$2\cdot8^{\circ}$	5·6	$6^{\circ}8^{\circ}$	7·4	$10^{\circ}8^{\circ}$	9·7	$14^{\circ}8^{\circ}$	12·5	$18^{\circ}8^{\circ}$	16·1
$2\cdot9^{\circ}$	5·6	$6^{\circ}9^{\circ}$	7·4	$10^{\circ}9^{\circ}$	9·7	$14^{\circ}9^{\circ}$	12·6	$18^{\circ}9^{\circ}$	16·2
$3\cdot0^{\circ}$	5·7	$7^{\circ}0^{\circ}$	7·5	$11^{\circ}0^{\circ}$	9·8	$15^{\circ}0^{\circ}$	12·7	$19^{\circ}0^{\circ}$	16·3
$3\cdot1^{\circ}$	5·7	$7^{\circ}1^{\circ}$	7·5	$11^{\circ}1^{\circ}$	9·9	$15^{\circ}1^{\circ}$	12·8	$19^{\circ}1^{\circ}$	16·4
$3\cdot2^{\circ}$	5·8	$7^{\circ}2^{\circ}$	7·6	$11^{\circ}2^{\circ}$	9·9	$15^{\circ}2^{\circ}$	12·9	$19^{\circ}2^{\circ}$	16·4
$3\cdot3^{\circ}$	5·8	$7^{\circ}3^{\circ}$	7·6	$11^{\circ}3^{\circ}$	10·0	$15^{\circ}3^{\circ}$	12·9	$19^{\circ}3^{\circ}$	16·7
$3\cdot4^{\circ}$	5·8	$7^{\circ}4^{\circ}$	7·7	$11^{\circ}4^{\circ}$	10·1	$15^{\circ}4^{\circ}$	13·0	$19^{\circ}4^{\circ}$	16·8
$3\cdot5^{\circ}$	5·9	$7^{\circ}5^{\circ}$	7·8	$11^{\circ}5^{\circ}$	10·1	$15^{\circ}5^{\circ}$	13·1	$19^{\circ}5^{\circ}$	16·9
$3\cdot6^{\circ}$	5·9	$7^{\circ}6^{\circ}$	7·8	$11^{\circ}6^{\circ}$	10·2	$15^{\circ}6^{\circ}$	13·2	$19^{\circ}6^{\circ}$	17·0
$3\cdot7^{\circ}$	6·0	$7^{\circ}7^{\circ}$	7·9	$11^{\circ}7^{\circ}$	10·3	$15^{\circ}7^{\circ}$	13·3	$19^{\circ}7^{\circ}$	17·1
$3\cdot8^{\circ}$	6·0	$7^{\circ}8^{\circ}$	7·9	$11^{\circ}8^{\circ}$	10·3	$15^{\circ}8^{\circ}$	13·4	$19^{\circ}8^{\circ}$	17·2
$3\cdot9^{\circ}$	6·1	$7^{\circ}9^{\circ}$	8·0	$11^{\circ}9^{\circ}$	10·4	$15^{\circ}9^{\circ}$	13·5	$19^{\circ}9^{\circ}$	17·3

## Elasticity of Aqueous Vapour—continued.

Tempera-ture C.	Tension in Min. Mercury.								
20° 0°	17·4	22° 0°	19·7	24° 0°	22·2	26° 0°	25·0	28·0	28·1
20° 4°	17·5	22° 1°	19·8	24° 1°	22·3	26° 1°	25·1	28·1	28·3
20° 2°	17·6	22° 2°	19·9	24° 2°	22·5	26° 2°	25·2	28·2	28·4
20° 3°	17·7	22° 3°	20·0	24° 3°	22·6	26° 3°	25·4	28·3	28·5
20° 4°	17·8	22° 4°	20·1	24° 4°	22·7	26° 4°	25·6	28·4	28·8
20° 5°	17·9	22° 5°	20·3	24° 5°	22·9	26° 5°	25·7	28·5	28·9
20° 6°	18·0	22° 6°	20·4	24° 6°	23·0	26° 6°	25·9	28·6	29·1
20° 7°	18·2	22° 7°	20·5	24° 7°	23·1	26° 7°	26·0	28·7	29·3
20° 8°	18·3	22° 8°	20·6	24° 8°	23·3	26° 8°	26·2	28·8	29·4
20° 9°	18·4	22° 9°	20·8	24° 9°	23·4	26° 9°	26·4	28·9	29·6
21° 0°	18·5	23° 0°	20·9	25° 0°	23·5	27° 0°	26·5	29·0	29·8
21° 1°	18·6	23° 1°	21·0	25° 1°	23·7	27° 1°	26·7	29·1	30·0
21° 2°	18·7	23° 2°	21·1	25° 2°	23·8	27° 2°	26·8	29·2	30·1
21° 3°	18·8	23° 3°	21·3	25° 3°	24·0	27° 3°	27·0	29·3	30·3
21° 4°	19·0	23° 4°	21·4	25° 4°	24·1	27° 4°	27·1	29·4	30·5
21° 5°	19·1	23° 5°	21·5	25° 5°	24·3	27° 5°	27·3	29·5	30·7
21° 6°	19·2	23° 6°	21·7	25° 6°	24·4	27° 6°	27·5	29·6	30·8
21° 7°	19·3	23° 7°	21·8	25° 7°	24·6	27° 7°	27·6	29·7	31·0
21° 8°	19·4	23° 8°	21·9	25° 8°	24·7	27° 8°	27·8	29·8	31·2
21° 9°	19·5	23° 9°	22·1	25° 9°	24·8	27° 9°	27·9	29·9	31·4

## Alcohol Table.

Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. under Proof.	Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. under Proof.
1·0000	0·00	0·00	100·00	0·9925	4·31	5·39	90·55
0·9995	0·26	0·33	99·42	0·9920	4·62	5·78	89·87
0·9990	0·53	0·66	98·84	0·9915	4·94	6·17	89·20
0·9985	0·79	0·99	98·26	0·9910	5·25	6·55	88·50
0·9980	1·06	1·34	97·66	0·9905	5·56	6·94	87·84
0·9975	1·37	1·73	96·97	0·9900	5·87	7·32	87·16
0·9970	1·69	2·12	96·29	0·9895	6·21	7·74	86·43
0·9965	2·00	2·51	95·60	0·9890	6·57	8·18	85·65
0·9960	2·28	2·86	95·00	0·9885	6·93	8·63	84·88
0·9955	2·56	3·21	94·40	0·9880	7·27	9·04	84·15
0·9950	2·83	3·55	93·78	0·9875	7·60	9·45	83·43
0·9945	3·12	3·90	93·16	0·9870	7·93	9·86	82·70
0·9940	3·41	4·27	92·50	0·9865	8·29	10·30	81·96
0·9935	3·71	4·63	91·87	0·9860	8·64	10·73	81·20
0·9930	4·00	5·00	91·23	0·9855	9·00	11·17	80·42

## Alcohol Table—continued.

Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. under Proof.	Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. under Proof.
0·9850	9·36	11·61	79·65	0·9610	27·93	33·81	40·74
0·9845	9·71	12·05	78·90	0·9605	28·25	34·18	40·10
0·9840	10·08	12·49	78·10	0·9600	28·56	34·54	39·47
0·9835	10·46	12·96	77·30	0·9595	28·87	34·90	38·84
0·9830	10·85	13·43	76·46	0·9590	29·20	35·28	38·18
0·9825	11·23	13·90	75·64	0·9585	29·53	35·66	37·50
0·9820	11·62	14·37	74·82	0·9580	29·87	36·04	36·83
0·9815	12·00	14·84	74·00	0·9575	30·17	36·39	36·23
0·9810	12·38	15·30	73·18	0·9570	30·44	36·70	35·68
0·9805	12·77	15·77	72·36	0·9565	30·72	37·02	35·13
0·9800	13·15	16·24	71·54	0·9560	31·00	37·34	34·57
0·9795	13·54	16·70	70·73	0·9555	31·31	37·69	33·95
0·9790	13·92	17·17	69·90	0·9550	31·62	38·04	33·32
0·9785	14·36	17·70	68·97	0·9545	31·94	38·46	32·70
0·9780	14·82	18·25	68·00	0·9540	32·25	38·75	32·08
0·9775	15·25	18·78	67·10	0·9535	32·56	39·11	31·46
0·9770	15·67	19·28	66·20	0·9530	32·87	39·47	30·84
0·9765	16·08	19·78	65·34	0·9525	33·18	39·81	30·24
0·9760	16·46	20·24	64·53	0·9520	33·47	40·14	29·66
0·9755	16·85	20·71	63·72	0·9515	33·76	40·47	29·08
0·9750	17·25	21·19	62·87	0·9510	34·05	40·79	28·52
0·9745	17·67	21·69	62·00	0·9505	34·29	41·05	28·06
0·9740	18·08	22·18	61·13	0·9500	34·52	41·32	27·60
0·9735	18·46	22·64	60·32	0·9495	34·76	41·59	27·13
0·9730	18·85	23·10	59·52	0·9490	35·00	41·84	26·67
0·9725	19·23	23·58	58·67	0·9485	35·25	42·18	26·20
0·9720	19·67	24·08	57·80	0·9480	35·50	42·42	25·70
0·9715	20·08	24·58	56·93	0·9475	35·75	42·67	25·22
0·9710	20·50	25·07	56·06	0·9470	36·00	42·95	24·74
0·9705	20·92	25·57	55·20	0·9465	36·28	43·26	24·20
0·9700	21·31	26·04	54·37	0·9460	36·56	43·56	23·66
0·9695	21·69	26·49	53·57	0·9455	36·83	43·87	23·12
0·9690	22·08	26·95	52·77	0·9450	37·11	44·18	22·58
0·9685	22·46	27·40	51·98	0·9445	37·39	44·49	22·04
0·9680	22·85	27·86	51·18	0·9440	37·67	44·79	21·50
0·9675	23·23	28·31	50·38	0·9435	37·94	45·10	20·96
0·9670	23·62	28·77	49·60	0·9430	38·22	45·41	20·43
0·9665	24·00	29·22	48·80	0·9425	38·50	45·71	19·89
0·9660	24·38	29·67	48·00	0·9420	38·78	46·02	19·36
0·9655	24·77	30·13	47·20	0·9415	39·05	46·32	18·83
0·9650	25·14	30·57	46·44	0·9410	39·30	46·59	18·36
0·9645	25·50	30·98	45·70	0·9405	39·55	46·86	17·88
0·9640	25·86	31·40	44·97	0·9400	39·80	47·13	17·40
0·9635	26·20	31·80	44·27	0·9395	40·05	47·40	16·93
0·9630	26·53	32·19	43·60	0·9390	40·30	47·67	16·46
0·9625	26·87	32·58	42·90	0·9385	40·55	47·94	15·98
0·9620	27·21	32·98	42·20	0·9380	40·80	48·21	15·50
0·9615	27·57	33·39	41·47	0·9375	41·05	48·48	15·04

## Alcohol Table—continued.

Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. under Proof.	Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. over Proof.
0·9370	41·30	48·75	14·57	0·9135	52·00	59·84	4·87
0·9365	41·55	49·02	14·10	0·9130	52·23	60·07	5·27
0·9360	41·80	49·29	13·63	0·9125	52·45	60·30	5·67
0·9355	42·05	49·55	13·16	0·9120	52·68	60·52	6·07
0·9350	42·29	49·81	12·70	0·9115	52·91	60·74	6·47
0·9345	42·52	50·06	12·27	0·9110	53·13	60·97	6·86
0·9340	42·76	50·31	11·82	0·9105	53·35	61·19	7·23
0·9335	43·00	50·57	11·38	0·9100	53·57	61·40	7·61
0·9330	43·24	50·82	10·94	0·9095	53·78	61·62	7·99
0·9325	43·48	51·07	10·50	0·9090	54·00	61·84	8·36
0·9320	43·71	51·32	10·05	0·9085	54·24	62·07	8·78
0·9315	43·95	51·58	9·60	0·9080	54·48	62·31	9·20
0·9310	44·18	51·82	9·20	0·9075	54·71	62·55	9·62
0·9305	44·41	52·06	8·77	0·9070	54·95	62·79	10·03
0·9300	44·64	52·29	8·36	0·9065	55·18	63·02	10·44
0·9295	44·86	52·53	7·94	0·9060	55·41	63·24	10·84
0·9290	45·09	52·77	7·52	0·9055	55·64	63·46	11·24
0·9285	45·32	53·01	7·10	0·9050	55·86	63·69	11·64
0·9280	45·52	53·24	6·70	0·9045	56·09	63·91	12·03
0·9275	45·77	53·48	6·27	0·9040	56·32	64·14	12·41
0·9270	46·00	53·72	5·86	0·9035	56·55	64·36	12·80
0·9265	46·23	53·95	5·45	0·9030	56·77	64·58	13·18
0·9260	46·46	54·19	5·03	0·9025	57·00	64·80	13·57
0·9255	46·68	54·43	4·62	0·9020	57·22	65·01	13·92
0·9250	46·91	54·66	4·20	0·9015	57·42	65·21	14·27
0·9245	47·14	54·90	3·80	0·9010	57·63	65·41	14·62
0·9240	47·36	55·13	3·38	0·9005	57·83	65·61	14·97
0·9235	47·59	55·37	2·97	0·9000	58·05	65·81	15·33
0·9230	47·82	55·60	2·56	0·8995	58·27	66·03	15·72
0·9225	48·05	55·83	2·15	0·8990	58·50	66·25	16·11
0·9220	48·27	56·07	1·74	0·8985	58·73	66·47	16·49
0·9215	48·50	56·30	1·33	0·8980	58·95	66·69	16·88
0·9210	48·73	56·54	0·92	0·8975	59·17	66·90	17·25
0·9205	48·96	56·77	0·50	0·8970	59·39	67·11	17·61
0·9200	49·16	56·98	0·14	0·8965	59·61	67·32	17·98
0·9198	49·24	57·06	Proof	0·8960	59·83	67·53	18·34
0·9195	49·39	57·20	0·25	0·8955	60·04	67·73	18·70
0·9190	49·64	57·45	0·68	0·8950	60·26	67·93	19·05
0·9185	49·86	57·69	1·10	0·8945	60·46	68·13	19·39
0·9180	50·09	57·92	1·51	0·8940	60·67	68·33	19·74
0·9175	50·30	58·14	1·89	0·8935	60·88	68·52	20·08
0·9170	50·52	58·36	2·28	0·8930	61·08	68·72	20·42
0·9165	50·74	58·58	2·66	0·8925	61·29	68·91	20·77
0·9160	50·96	58·80	3·05	0·8920	61·50	69·11	21·11
0·9155	51·17	59·01	3·41	0·8915	61·71	69·30	21·45
0·9150	51·38	59·22	3·78	0·8910	61·92	69·50	21·79
0·9145	51·58	59·43	4·14	0·8905	62·14	69·71	22·16
0·9140	51·79	59·63	4·50	0·8900	62·36	69·92	22·53

## Alcohol Table—continued.

Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. over Proof.	Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. over Proof.
0·8895	62·59	70·14	22·91	0·8655	72·74	79·31	38·99
0·8890	62·82	70·35	23·29	0·8650	72·96	79·50	39·32
0·8885	63·04	70·57	23·66	0·8645	73·17	79·68	39·64
0·8880	63·26	70·77	24·02	0·8640	73·38	79·86	39·96
0·8875	63·48	70·97	24·37	0·8635	73·58	80·04	40·27
0·8870	63·70	71·17	24·73	0·8630	73·79	80·22	40·60
0·8865	63·91	71·38	25·09	0·8625	74·00	80·40	40·91
0·8860	64·13	71·58	25·44	0·8620	74·23	80·60	41·26
0·8855	64·35	71·78	25·79	0·8615	74·45	80·80	41·61
0·8850	64·57	71·98	26·15	0·8610	74·68	81·00	41·96
0·8845	64·78	72·18	26·50	0·8605	74·91	81·20	42·31
0·8840	65·00	72·38	26·85	0·8600	75·14	81·40	42·66
0·8835	65·21	72·58	27·19	0·8595	75·36	81·60	43·00
0·8830	65·42	72·77	27·52	0·8590	75·59	81·80	43·35
0·8825	65·63	72·96	27·85	0·8585	75·82	82·00	43·70
0·8820	65·83	73·15	28·19	0·8580	76·04	82·19	44·04
0·8815	66·04	73·34	28·52	0·8575	76·25	82·37	44·35
0·8810	66·26	73·54	28·87	0·8570	76·46	82·54	44·66
0·8805	66·48	73·73	29·22	0·8565	76·67	82·70	44·97
0·8800	66·70	73·93	29·57	0·8560	76·88	82·90	45·28
0·8795	66·91	74·13	29·92	0·8555	77·08	83·07	45·60
0·8790	67·13	74·33	30·26	0·8550	77·29	83·25	45·90
0·8785	67·33	74·52	30·59	0·8545	77·50	83·43	46·20
0·8780	67·54	74·70	30·92	0·8540	77·71	83·60	46·51
0·8775	67·75	74·89	31·25	0·8535	77·92	83·78	46·82
0·8770	67·96	75·08	31·58	0·8530	78·12	83·98	47·11
0·8765	68·17	75·27	31·90	0·8525	78·32	84·11	47·40
0·8760	68·38	75·45	32·23	0·8520	78·52	84·27	47·70
0·8755	68·58	75·64	32·56	0·8515	78·72	84·44	47·98
0·8750	68·79	75·83	32·89	0·8510	78·92	84·60	48·27
0·8745	69·00	76·01	33·21	0·8505	79·12	84·77	48·56
0·8740	69·21	76·20	33·54	0·8500	79·32	84·93	48·84
0·8735	69·42	76·39	33·86	0·8495	79·52	85·10	49·13
0·8730	69·63	76·57	34·19	0·8490	79·72	85·26	49·38
0·8725	69·83	76·76	34·51	0·8485	79·92	85·42	49·67
0·8720	70·04	76·94	34·84	0·8480	80·13	85·59	50·00
0·8715	70·24	77·12	35·14	0·8475	80·33	85·77	50·31
0·8710	70·44	77·29	35·45	0·8470	80·54	85·94	50·61
0·8705	70·64	77·46	35·76	0·8465	80·75	86·11	50·91
0·8700	70·84	77·64	36·07	0·8460	80·96	86·28	51·21
0·8695	71·04	77·82	36·37	0·8455	81·16	86·45	51·50
0·8690	71·25	78·00	36·69	0·8450	81·36	86·61	51·78
0·8685	71·46	78·18	37·01	0·8445	81·56	86·77	52·06
0·8680	71·67	78·36	37·33	0·8440	81·76	86·93	52·34
0·8675	71·88	78·55	37·65	0·8435	81·96	87·09	52·62
0·8670	72·09	78·73	37·98	0·8430	82·15	87·24	52·90
0·8665	72·30	78·93	38·32	0·8425	82·35	87·40	53·16
0·8660	72·52	79·12	38·65	0·8420	82·54	87·55	53·43

Alcohol Table—*continued.*

Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. over Proof.	Specific Gravity at 60° F.	Per Cent. of Alcohol by Weight.	Per Cent. of Alcohol by Volume.	Per Cent. over Proof.
0·8415	82·73	87·70	53·70	0·8170	92·07	94·76	66·07
0·8410	82·92	87·85	53·96	0·8165	92·26	94·90	66·30
0·8405	83·12	88·00	54·23	0·8160	92·44	95·03	66·53
0·8400	83·31	88·16	54·50	0·8155	92·03	95·16	66·76
0·8395	83·50	88·31	54·75	0·8150	92·81	95·29	67·00
0·8390	83·69	88·46	55·02	0·8145	93·00	95·42	67·23
0·8385	83·88	88·61	55·28	0·8140	93·18	95·55	67·46
0·8380	84·08	88·76	55·55	0·8135	93·37	95·69	67·70
0·8375	84·28	88·92	55·83	0·8130	93·55	95·82	67·92
0·8370	84·48	89·08	56·10	0·8125	93·74	95·95	68·15
0·8365	84·68	89·24	56·38	0·8120	93·92	96·08	68·38
0·8360	84·88	89·39	56·66	0·8115	94·10	96·20	68·60
0·8355	85·08	89·55	56·93	0·8110	94·28	96·32	68·80
0·8350	85·27	89·70	57·20	0·8105	94·45	96·43	69·00
0·8345	85·46	89·84	57·45	0·8100	94·62	96·55	69·20
0·8340	85·65	89·99	57·71	0·8095	94·80	96·37	69·40
0·8335	85·85	90·14	57·97	0·8090	94·97	96·78	69·61
0·8330	86·04	90·29	58·23	0·8085	95·14	96·90	69·82
0·8325	86·23	90·43	58·48	0·8080	95·32	97·02	70·03
0·8320	86·42	90·58	58·74	0·8075	95·50	97·15	70·25
0·8315	86·62	90·73	59·00	0·8070	95·68	97·27	70·46
0·8310	86·81	90·88	59·26	0·8065	95·86	97·39	70·67
0·8305	87·00	91·02	59·51	0·8060	96·03	97·51	70·88
0·8300	87·19	91·17	59·77	0·8055	96·20	97·62	71·07
0·8295	87·38	91·31	60·02	0·8050	96·37	97·73	71·26
0·8290	87·58	91·46	60·28	0·8045	96·53	97·83	71·45
0·8285	87·77	91·60	60·53	0·8040	96·70	97·94	71·64
0·8280	87·96	91·75	60·79	0·8035	96·87	98·05	71·83
0·8275	88·16	91·90	61·05	0·8030	97·03	98·16	72·02
0·8270	88·36	92·05	61·32	0·8025	97·20	98·27	72·40
0·8265	88·56	92·21	61·60	0·8020	97·37	98·37	72·58
0·8260	88·76	92·36	61·86	0·8015	97·53	98·48	72·77
0·8255	88·96	92·51	62·12	0·8010	97·70	98·59	72·95
0·8250	89·16	92·66	62·38	0·8005	97·87	98·69	73·14
0·8245	89·35	92·80	62·63	0·8000	98·03	98·80	73·30
0·8240	89·54	92·94	62·88	0·7995	98·19	99·89	73·47
0·8235	89·73	93·09	63·13	0·7990	98·34	99·98	73·64
0·8230	89·92	93·23	63·38	0·7985	98·50	99·07	73·81
0·8225	90·11	93·36	63·62	0·7980	98·66	99·16	73·97
0·8220	90·29	93·49	63·84	0·7975	98·81	99·26	74·14
0·8215	90·46	93·62	64·06	0·7970	98·97	99·35	74·31
0·8210	90·64	93·75	64·30	0·7965	99·13	99·45	74·50
0·8205	90·82	93·87	64·51	0·7960	99·29	99·55	74·66
0·8200	91·00	94·00	64·74	0·7955	99·45	99·65	74·83
0·8195	91·18	94·13	64·96	0·7950	99·61	99·75	75·01
0·8190	91·36	94·26	65·18	0·7945	99·78	99·86	75·18
0·8185	91·54	94·38	65·40	0·7940	99·94	99·96	75·25
0·8180	91·71	94·51	65·62	0·7938	100·00	100·00	
0·8175	91·89	94·64	65·85				

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